



# ***JPRS Report***

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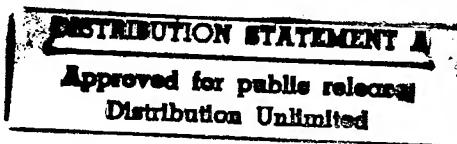
# **Science & Technology**

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***USSR: Chemistry***

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# Science & Technology

## USSR: Chemistry

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31 January 1991

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## RADIATION CHEMISTRY

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UDC 541.183.5

**Effect of the Adsorption and Electrosurface Properties of Membranes Modified with Chloride Alkylpyridines on Degree of Separation**

907D0291A Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 600-603

[Abstract of article by N. A. Yaroshenko, N. V. Vdovenko; Institute of Colloidal and Water Chemistry imeni A. V. Dumanskiy, Ukrainian Academy of Sciences, Kiev]

[Abstract] The adsorption of homologous series of chloride N-alkylpyridines from aqueous and 0.2 M NaCl solutions by an MK-40 cationic membrane at 20°C was studied under equilibrium conditions. The solid phase concentration was 0.01-0.2% for the higher homologous compounds of the tetradecyl 1 and hexadecyl 1 series, and 0.01-1% for the decyl 1 and dodecyl 1 series. The specific surface of the MK-40 membrane was calculated to be 85 m<sup>2</sup>/g, which is very close to that of cellulose acetate membranes. Maximum adsorption was 3.5 to 25.7 times greater for the MK-40 than for a representative cellulose acetate membrane and was attributed to larger pore size. The highest values were observed for the tetra- and hexadecyl 1 series in water. For all series, however, maximum adsorption was considerably lower than the exchange capacity of the membrane. The foregoing, along with the electrokinetic and electrosurface properties of the membrane, was used to construct a model of the adsorption layer. In this model, the alkylpyridine ions are exchanged for hydrogen cations, and the membrane surface becomes hydrophobic, hence the reduced membrane exchange capacity. As the surfactant concentration is increased, the hydrophobic radicals of the orienting hydrophobic groups interact dispersively with the first adsorbed monolayer. For all the homologous series, saturation at the membrane/solution boundary is 0.5 mmol/l. In contrast to cellulose acetate membranes, capillary "superconductivity" was observed in the MK-40 membrane, and, in micellar solutions, the membrane was partially "poisoned" and its selectivity degraded. Figures 2, tables 1; references 14: 12 Russian, 2 Western.

UDC 541.335.21:546.215

**Study of the Formation and Stability of Hydrogen Peroxide in Electrolyzed Solutions**

907D0291B Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 607-609

[Abstract of article by S. I. Agadzhanyan, S. M. Soryanova, G. V. Korshin, A. G. Liakumovich, R. G. Romanova, O. I. Rusetskiy; Kazan Chemical Technology Institute imeni S. M. Kirov]

[Abstract] The formation and stability of hydrogen peroxide in electrolyzed solutions was studied. Hydrogen peroxide concentration was determined spectrophotometrically and polarographically. Oxygen content was measured with an ELWRO-5113 amperometric sensor. Electrolysis was performed for 20 to 30 min in a diaphragm-equipped electrolytic cell with graphite or platinum

electrodes and flowing or stationary electrolyte. Current density varied from 0.2 to 1 A/dm<sup>2</sup>; from 0 to 900 coul/l of electricity was passed through unit volume of solution. The electrolyzed solutions consisted of 10<sup>-3</sup> to 10<sup>-2</sup> M K<sub>2</sub>SO<sub>4</sub> or 0.14 M NaCl prepared over distilled water. An increase in current potential to -1.5 V in deoxygenated solution was taken as an indication of the presence of hydrogen peroxide. Polarograms of the catholyte and anolyte did not reveal the presence of hydrogen peroxide. In those few cases where the polarograms indicated current potential approaching -1.5 V, spectrophotometric analysis showed no hydrogen peroxide, and the potential value was attributed to other factors. When a known quantity (10<sup>-3</sup> to 5(10<sup>-2</sup>)) of hydrogen peroxide was added to the original solution, electrolysis resulted in a drop in hydrogen peroxide content, and the longer the process, the faster the drop. Hydrogen peroxide did not form in quantities exceeding 10<sup>-5</sup> mol/l, and its presence could not be attributed to electrolytic processes. Therefore, certain properties acquired by solutions during electrolysis cannot be attributed to hydrogen peroxide formation or behavior. Figures 4; references 14: 12 Russian, 2 Western.

UDC 66.067.32

**Structure and Selectivity of Modified Ceramic Filters**

907D0291C Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 610-612

[Abstract of article by I. Ye. Skobets, Ye. A. Serpuchenko; Institute of Colloidal and Water Chemistry imeni A. V. Dumanskiy, Ukrainian Academy of Sciences, Kiev]

[Abstract] A study was done to determine whether the applications of existing ceramic filters could be increased by metallizing them. Ceramic material from the Khutsk ceramics plant was used as the substrate. The filters used in the study had identical productivity and porosity specifications. A layer of copper was deposited on the ceramic substrate, using formaldehyde to induce a reduction reaction of copper from copper salts in an alkaline medium. After the specimens were coppered, they were rinsed, dried at 375 K, and annealed in a muffle furnace at 670 K. Optimum coppering time was two hours, during which a coating consisting of copper and copper oxides that tenaciously adhered to the filter surface formed. No copper ions were detected by NMR on the coating surface. Coppering did not change the general nature of pore distribution by size, but overall porosity and productivity decreased. The number of pores 0.1 to 0.3 μm in diameter increased, while the number of pores 0.5 to 1.5 μm in diameter decreased. The transport pores decreased in size from 1.5 to 0.3 μm. The relationship between structural and filtration characteristics was studied by performing filtering polyethylene glycols with molecular masses of 20, 40, 60, 150, and 650 thousand. The metallized filters were completely permeable to all the influents with a molecular mass below 650,000. There was a gradual decrease in productivity as molecular mass increased. The study showed that the transport selectivity of ceramic filters can

be controlled by metallizing them, thereby increasing their range of applications. Figures 3, tables 1; references 7: Russian.

UDC 615.838.7.074:546.815/819:541.183:535.24

### Photometric Determination of Sorption Concentrated Lead in Natural Objects

907D0291D Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 627-629

[Abstract of article by N. M. Malakhova, Ye. M. Nikipelova, G. I. Savenko; Odessa State University imeni I. I. Mechnikova, Odessa Scientific Research Institute of Health Resort Sciences and Convalescence\*\*\*]

[Abstract] The sorption precipitation of lead (II) from aqueous solutions with subsequent photometric analysis was studied. A model solution was made by mixing 1.5 g of air-dried Crimean mud with hydrofluoric and sulfuric acids. After the sample was treated with HCl and distilled water, 5 ml of the solution were put into a beaker. One ml of a 10%-solution of ascorbic acid and 0.9 ml of a 0.1 M solution of ammonium difluoride were added as masking agents to prevent chromium, aluminum, iron, copper, and cobalt ions from hindering maximum lead desorption. The pH was brought to 8.0, and enough water added to obtain 25 ml of solution, which was then transferred to a 50-ml flask containing SG 5/40 silica gel, which had the highest degree of sorption of the three silica gels evaluated. The flask was shaken for 60 min on an AVU-6 automatic shaker. The sorbent was separated centrifugally from the solution in 5 min, rinsed with distilled water, and the lead desorbed with 10 ml of a 0.1 M HCl solution. To determine the lead concentration, the precipitate was transferred to another beaker, the pH set at 4, and 10 ml of borate buffer added. This solution was transferred to a flask, 2 ml of a  $1(10^{-3})$  M aqueous solution of 1-(2-pyridylas)-resorcinol added to it, topped up [presumably with precipitate]\*\*\*, and stirred. The optical density of the solution was measured relative to a dummy test solution by an SF-16 spectrophotometer in a cell 2 cm long at 540 nm. Lead concentration was determined graphically. The results very closely approximated the results from an atomic-emission analysis of lead content. This method can be used to detect lead concentrations as low as 0.01  $\mu\text{g}/\text{ml}$  of lead in therapeutic muds, waters, and so forth. Tables 3; references 6: 5 Russian, 1 Western.

UDC 621.359.7

### Use of MK-40 and MA-40 Heterogeneous Membranes in the Electrodialysis of Manganese-Containing Solutions

907D0291E Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 630-632

[Abstract of article by L. A. Melnik, B. K. Melyayeva, V. D. Grebenyuk, Kh. N. Yevzhanov; Institute of Colloidal and Water Chemistry imeni A. V. Dumanskiy, Ukrainian Academy of Sciences, Kiev]

[Abstract] The transport of manganese compounds through heterogeneous MK-40 and MA-40 membranes at various pH levels was studied. The tests were performed in a five-chamber electrodialyzer built according to the following configuration: platinum cathode—electrode chamber—MA-40—concentration chamber—MK-40—desalination chamber—MA-40—concentration chamber—MK-40—electrode chamber—platinum anode. Each membrane had a working surface of 9.7 sq cm, and the distance between membranes was 5 mm. In the desalination chamber, 5 cu dm of a 0.2 M NaCl solution containing 10 mg/cu dm of manganese ( $\text{MnCl}_2$ ) circulated at a linear speed of 5 cm/s. The solution pH was varied from 2.0 to 11.0 and monitored by an EV- 74. A 0.1 M sodium sulfate solution was pumped through the electrode chambers. Manganese transfer was evaluated from samples taken periodically from the concentration chamber. Manganese content was measured on a Pye-Unicam atomic absorber. There was no transfer of iron or manganese compounds through the MA-40 membrane at any pH level. Transfer of manganese through the MK- 40 membrane was substantial when pH was from 2 to 8, but tapered off sharply at higher pH levels. The relatively short transfer time (3-5) hours indicates the sufficiently high mobility of the  $\text{Mn}^{2+}$  ion. Transfer does not occur in highly alkaline solutions because the  $\text{Mn}^{2+}$  ion is oxidized to a higher valence state by the oxygen dissolved in the water, and poorly soluble compounds are formed. Iron transfer through the MK-40 was minimal or non-existent at pH levels greater than 4 for many of the same reasons. Figures 3; references 17: 15 Russian, 2 Western.

UDC 628.036

### Using Clinomordenite to Defluorinate Natural Waters

907D0291F Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 647-649

[Abstract of article by V. A. Kravchenko, N. D. Kravchenko, G. G. Rudenko, Yu. I. Tarasevich; Scientific Research and Industrial Design Institute of Public Utilities of the Ukrainian Minzhilkomkhoz and Institute of Colloidal and Water Chemistry imeni A. V. Dumanskiy of the Ukrainian Academy of Sciences, Kiev]

[Abstract] A special pilot unit was designed and tested to determine the feasibility of using clinomordenite pre-modified with aluminum sulfate to defluorinate water. A special series of experiments using various filter materials showed that the clinomordenite was by far the best fluoride ion sorbent. The unit's operations can be divided into five stages. First, the filter material is crumbled and rinsed with water fed from bottom to top at 15 to 17 cu dm/(c by sq m). The sorbent is then modified by passing a 0.5-% solution of coagulant and aluminum sulfate through it from top to bottom at a rate of 10-15 m/h for 1 to 1.5 h. Surplus modifying solution is then rinsed away using the starting fluorinated water. This stage is finished when the aluminum concentration in the filtrate is reduced to 0.5 mg/cu dm. It also lasts 1 to 1.5 h. The fourth stage is the actual defluorination process, followed by removal of the fluoride ions. A two-fold increase in filtration speed

reduced defluorination time two-fold. However, the sorbent had a greater defluorination capacity at lower filtration speeds. With this method, defluorination primarily occurs as a result of ion-exchange displacement/substitution\*\*\* from the surface of the granular composition consisting of clinomordenite, the products of the hydrolysis of the coagulant, and the hydroxyl and sulfate ions by the fluoride ions of the treated water. To prolong the defluorination process and conserve process water, water used to rinse away excess modifier is added to the water to be treated. Projected productivity of the unit is 10,000 to 22,600 cu m/24 h. Figures 5; references 4: 3 Russian, 1 Western.

UDC 579.6.695

**Transformation of Diethylene Glycol by Microorganisms in a Multisection Bioreactor**

907D0291G Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 652-654

[Abstract of article by P. I. Gvozdyak, L. I. Nesynova, V. M. Udod, G. S. Vengzhen, N. I. Podorvan, L. V. Nevinaya; Institute of Colloidal and Water Chemistry imeni A. V. Dumanskiy, Ukrainian Academy of Sciences, Kiev]

[Abstract] DEG transformation and utilization was modeled in an 18-section direct-flow bioreactor with a total capacity of 3.6 cu dm. Each section contained 5 g of polypropylene fiber and was aerated by an AEN-2 micro-compressor supplying an average of 10 cu dm of air per hour. The representative waste water was pumped through the reactor by an MP-1M peristaltic pump. The water contained, in g/cu dm: 0.6 Na<sub>2</sub>HPO<sub>4</sub>, 0.4 NH<sub>4</sub>NO<sub>3</sub>, 0.02 KCl, 0.01 MnSO<sub>4</sub>, 0.02 MgCl<sub>2</sub>, and 1, 2, or 3 of DEG. A suspension of active DEG-degrading organisms was introduced into the first section. After 10 days, the fibers hosted a biomass of microorganisms. Waste water was then pumped through the system, allowing the bacteria to be distributed throughout all sections of the reactor. The flow rate was stabilized at 0.04 h<sup>-1</sup>. Mass concentration of DEG and its transformation products were monitored on a Tsvet-152 gas chromatograph, using different extraction methods depending on the DEG mass fraction. The quantity of bacteria in a section was monitored microscopically from dry masses of cells washed from the fibers and amounted to 10%cu cm. The tests were carried out in each section for each quantity of DEG. The data showed that DEG in concentrations up to 3 g/cu dm is completely degraded by the bacteria and its transformation is accompanied by the formation of ethylene glycol, ethanol, and ascetic acid within the first five sections. Variation in the pH level, dehydrogenase activity, and phosphorus and nitrogen consumption indicate that several groups of microorganisms participate in DEG degradation through the spatial succession of two trophic levels: bacterial and a protozoan. Figures 3; references 17: 14 Russian, 3 Western.

UDC 628.3:577.153.1

**Study of the Feasibility of Using Lipases Immobilized on Carbon Mineral Sorbents to Treat Fat-Containing Waste Waters**

907D0291H Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 655-657

[Abstract of article by L. A. Fedoseyeva, S. Yu. Burylin, V. D. Sokolovskiy, L. N. Rachkovskaya, T. I. Politova, I. M. Arends, I. I. Frolova; Institute of Catalysis of the Siberian Division of the Ukrainian Academy of Sciences, Novosibirsk]

[Abstract] A study was done to determine whether lipases immobilized on carbon mineral sorbents could be used to remove fats from waste waters. Six different sorbents were impregnated with lipoorizine solutions varying in concentration from 5 to 100 mg/ml to produce biocatalysts, which were rinsed with a buffer solution to remove unsorbed enzyme. Lipase activity (LA), which characterizes the ability of the enzyme to catalyze the breakdown of an olive oil emulsion, was then measured. To do this, a mixture of 2.5 ml of 40% olive oil emulsion, 2 ml of Makilvein\*\*\* citrate-phosphate buffer with pH 7, and 0.5 ml of water preheated at 37°C for 30 min was added to 1 g of sorbent, which was then incubated for 1 h at 37°C while being continually stirred in a thermostatically controlled mixer. The mixture was then poured into a flask with 15 ml of ethanol, the catalyst rinsed with 5 ml of water, and the rinse water poured into the same flask. Titration and calculations were performed using previously described methods. (The measurements are expressed in lipase units (le)/g of sorbent, and correspond to the liberation of 1 μmol of oleic acid in one hour at 37°C.) The GZKh lipoorizine used to prepare the catalysts had an LA of 180,000. All but one of the sorbents fully adsorbed the lipase solution, and LA varied from 75 to 250 le/g, depending on the sorbent used. LA decreased by no more than 50% when the biocatalysts were used in conditions simulating those of waste waters from meat processing plants. When immersed in actual waste waters from meat processing plants, biocatalyst LA for 34 days remained as high as that of biocatalysts immersed in city water supply mains. Tables 1; references 7: 5 Russian.

UDC 628.33:628.315/317

**On the Mechanism By Which Coli Bacteria is Deactivated During the Electrical Decontamination of Water**

907D0291I Kiev KHIMIYA I TEKHOLOGIYA VODY in Russian Vol 12, No 7, Jul 90 pp 658-661

[Abstract of article by L. I. Markitanova, G. S. Zenin; Leningrad Technical Institute of Refrigeration]

[Abstract] A study was done to determine how certain factors affect the electrical decontamination of water. The effect of the redox potential of "activated" water on the viability of coli bacteria was tested by electrolyzing a 0.001-0.25 mol/l sodium sulfate solution for 100 s in a cell with a BS-7 lavsan\*\*\* fabric diaphragm separating the

graphite anode and cathodes. Up to  $10^7$  col/l of coli bacteria were than added to the anolyte and catholyte, and the redox potential and coli index measured. The data showed that "activated" water does not deactivate coli bacteria under the conditions tested. The effect of current density and electric field intensity on bacteria viability was tested by treating the solutions in a three-chamber flow-through electrolyzer with MA-40 and MK-40 ion-exchange membranes separating the chambers. (In both tests, a pH-242 meter and a KSP-4 recording potentiometer were used to record pH in the electrode cells, in the area in between, and in the anolyte and catholyte currents. Solution temperature was measured by a thermometer with an

accuracy of  $\pm 0.01^{\circ}\text{C}.$ ) This test showed that the coli bacteria are destroyed by coming in direct contact with the electrodes and reach them by electrical transfer and diffusion. The electrical field itself, within the range of intensities studied, does not affect the bacteria. The study showed that the laws governing electrical decontamination are subordinate to the relationships characterizing electrofiltration in a slotted channel. For aqueous solutions, a correlation was obtained that can be used to calculate the energy required for electrical decontamination depending on the saline concentration of the solution and the productivity of the electrolyzer. Figures 4, tables 1; references 10: Russian.

UDC 539.236

**Production of  $Al_2O_3$  Coatings by Thermal Dissociation of Gaseous Mixture of  $AlCl_3$ - $C_2H_3OH$**   
907M0243A Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 35,  
No 5, May 1990 pp 1134-1136

[Article by A. V. Orlov, A. N. Degtyarev and Yu. M. Grigoryev; Institute of Structural Macrokinetics; USSR Academy of Sciences]

[Abstract] A search for the cheapest and safest system for  $Al_2O_3$  deposition by the gas phase method involved an attempt to produce coatings by use of an  $AlCl_3$  system.  $Al_{23}$  films were produced on tungsten wire by thermal dissociation of a gaseous mixture of an  $AlCl_2Cl_3$  mixture. The dissociation process and the composition of the products formed were complex and not completely understood but depended upon both the nature of the aluminum-containing compounds and the deposition procedure. At a temperature below 700°C, the coating did not form or was deposited very slowly. Rate of deposition increased with a temperature increase and increased greatly in the 1050-1200°C range. Deposition on the wire did not occur at temperatures above 1200°C. The samples produced consisted of a mixture of several phases of  $\gamma$ ,  $\delta$  and  $\alpha=Al_2O_3$  with inclusions of aluminum and tungsten carbides. Figures 2; references 3: 1 Russian; 2 Western.

UDC 546.763.732'224

**Phase Transformations of  $CdCr_2Se_4$  at High Pressures and Temperatures**  
907M0243B Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 35,  
No 5, May 1990 pp 1137-1141

[Article by A. V. Filatov, Ye. A. Volkov, V. A. Davydov et al.; Institute of General and Inorganic Chemistry imeni N. S. Kurnakov; USSR Academy of Sciences; Institute of Physics of High Pressures imeni L. F. Vereshchagin, USSR Academy of Sciences]

[Abstract] A study of the effect of high pressure (0.5-3 GPa) and temperatures in the 500-1000°C range on the phase composition and physical properties of the magnetic semiconductor  $CdCr_2Se_4$  was described and discussed. Roentgenophase data, magnetic phase analysis and measurements of density and electroconductivity made it possible to construct the p-T-diagram of  $CdCr_2Se_4$  in the indicated pressure and temperature range. After hot pressing, the spinel modification was decomposed into  $Cr_2Se_3$  and  $CdSe$  at lower pressures and temperatures than are necessary for polymorphic transformation of  $CdCr_2$  in monocline modification. The dependence of the temperature of peritectoid decomposition of  $CdCr_2Se_4$  on the level of pressure applied was plotted on a p-T diagram. Figures 3; references 2 (Western).

UDC 546.74'72'34-31:547.67

**Magnetic Properties in  $LiFeO_2$ - $LiNiO_2$  System**  
907M0243C Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 35,  
No 5, May 1990 1267-1272

[Article by D. G. Kellerman, V. S. Gorshkov, V. A. Perelyayev and G. P. Shveykin; Institute of Chemistry, USSR Academy of Sciences UrO, Sverdlovsk]

[Abstract] The study examined the dependence of magnetic properties of solid solutions of an  $LiNiO_2$ - $LiFeO_2$  system on the degree of structural order. The region of compositions with cubical structure and the region of ordered composition were discussed separately. The nature of magnetic ordering was discussed. Figures 6; references 7: 3 Russian; 4 Western.

UDC 546.631+536.424.1

**Copper (III) in Complex Oxides  $LnBa_2Cu_3O_{7-y}$**   
907M0243D Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 35,  
No 5, May 1990 pp 1333-1334

[Article by I. S. Shaplygin, A. K. Misyavichus, I. A. Konovalova and V. B. Lazarev]

[Abstract] Determination of copper (III) in high temperature superconductors  $LnBa_2Cu_3O_{7-y}$  with  $Ln=Y$ , Nd, Eu, Gd, Ho was performed by iodometric titration with subsequent binding of the iodine by sodium thiosulfate. The mean value of  $Cu^{III}$  in samples studied was 24 + or - 2 percent of the total copper content. A discrepancy in analytical determinations of copper (III) even for type I oxides with similar oxygen content was attributed to redox processes. Reference 1 (Western).

UDC 541.1:546.65'131

**Temperature of Congruent Melting of Europium Trichloride**  
907M0243E Moscow ZHURNAL  
NEORGANICHESKOY KHIMII in Russian Vol 35,  
No 5, May 1990 pp 1334-1336

[Article by T. V. Kiseleva, V. F. Goryushkin and D. M. Laptev; Siberian Metallurgical Institute]

[Abstract] An attempt to determine the melting point of europium trichloride under conditions of its congruent melting used europium trichloride obtained from  $Eu_2O_3$  oxide by carbon tetrachloride chlorination. The melting point was determined by the differential thermal analysis with use of calcinated  $Al_2O_2$ . The temperature of congruent melting of  $EuCl_3$  was 632 + or - 7°C. Figure 1; references 7: 6 Russian; 1 Western.

UDC 541.49+546.562

**Magnetic Properties of Binuclear and Mononuclear Metal-chelates of Bivalent Copper With Aroylhydrazones of 2-acetonilbenzimidazole**  
*907M0243F Moscow Zhurnal NEORGANICHESKOY KHIMII in Russian Vol 35, No 5, May 1990 pp 1336-1337*

[Article by V. V. Lukov, V. A. Kogan, Ye. V. Bogatyreva and A. L. Yepifantsev; Rostov-on-Don State University]

[Abstract] A study of the magnetic properties of new binuclear and mononuclear metal-chelates based on aroylhydrazones of 2-acetonilbenzimidazole in the 80-300 K range provided an assumption concerning a possible cause of weak exchange interactions in these systems. Figure 1; references 3: 2 Russian; 1 Western.

UDC 546.19'23+657'23

**Constitution Diagram of  $As_2Se_3$ -NdSe System**  
*907M0243G Moscow ZHURNAL NEORGANICHESKOY KHIMII in Russian Vol 35, No 5, May 1990 pp 1353-1355*

[Article by G. P. Rustamov, T. M. Ilyasov and A. I. Nadzhafov]

[Abstract] Study of the  $As_2Se_3$ -NdSE section of a ternary system of Nd-As-Se by methods of physico-chemical analysis revealed a region of vitrification reaching up to 13 molar percent of NdSe. Investigation of the crystallization capacity of the glasses was described and discussed. A constitution diagram of the  $As_2Se_3$ -NdSe system was constructed and illustrated. On the basis of initial components were detected regions of solubility reaching up to 1.5 molar percent on the basis of  $As_2Se_3$  and 3 molar percent on the basis of NdSE. Figure 1; references 7: 6 Russian; 1 Western.

UDC 537.311.33

**Chemically Bound Complexes Involving Rapidly Diffusing Impurities in Silicon**  
*917M0022 Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 10, Oct 90 (manuscript received 10 Nov 88) pp 2005-2008*

[Article by M.K. Bakhadyrkhonov, Sh.I. Askarov, and N. Narkulov, Tashkent Polytechnic Institute imeni Beruni]

[Abstract] Monocrystalline silicon always contains some concentration of uncontrollable, rapidly diffusing impurities (such as Cu, Fe, or Cr) whose states in the lattice are unstable. During the process of the manufacture of semiconductor instruments, the material is subjected to different heat treatment, in which case those rapidly diffusing impurities participate in the formation of various heat donors and defects and diminish the parameters of the finished instruments. In view of this, it is critical that the rapidly diffusing impurities in the bulk of the crystal be

neutralized. In an attempt to refine the results obtained in earlier research, the authors of this study examined the formation of electrically neutral chemically bound complexes of sulfur with other rapidly diffusing impurities in silicon, specifically iron and chromium, and determined the technological conditions during which effective complexing occurs. A batch of sulfur-doped silicon specimens were studied at 1,250°C for 100 hours. A grade KDB-10 silicon monocrystal with the dimensions 10 x 4 x 2 mm was used as the starting material. The doping resulted in overcompensated Si[B, S] specimens with a value of  $\rho$  equal to or about 1  $\Omega\cdot cm$ . Next, the Si[B, S] specimens were doped with iron or chromium by diffusion annealing in the interval from 1,100 to 1,250°C. Control specimens of Si[B, S], Si[B, Fe], and Si[B, Cr] were also annealed to consider the effect of repeated annealing and to compare the electrical parameters in each case. It was found that regardless of the temperature, the parameters of the Si[B, S, Fe] and Si[B, S, Cr] specimens were comparable with those of the Si[B, S] controls. In the case of the Si[B, Fe] and Si[B, Cr] specimens, it was observed that as the annealing temperature increases,  $\rho$  increases, and subsequent overcompensation occurs at temperatures above 1,200°C to n-type conduction. These results confirm the predominantly donor nature of both iron and chromium in silicon. Next, to shed more light on the comparability of the parameters of Si[B, S, Cr] and Si[B, S, Fe] specimens with those of Si[B, S] specimens, all three types were subjected to 1 hour of annealing in the temperature range from 820 to 960°C. Neutralization of the electrically active impurity centers was observed afterward. The presence of a correlation between the temperature of the most effective neutralization of the impurities and the thermodynamic Gibbs energy of the respective compounds of these elements was established. It was concluded that the observed neutralization of the impurity centers results from the formation of electrically neutral chemically bound complexes between the sulfur substitution centers and the centers of the introduction of rapidly diffusing impurities. Tables 2; references 8: 6 Russian, 2 Western.

UDC 539.216.2:621.315.592

**Investigation of  $Ge_xSi_{1-x}$  Heteroepitaxial Structures Produced in a Vacuum**

*917M0022B Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 10, Oct 90 (manuscript received 10 Oct 88) pp 2009-2012*

[Article by L.N. Abrosimova, Yu.N. Drozdov, Gorkiy State University imeni N.I. Lobachevskiy, T.S. Kuntsevich, and V.A. Tolomasov, Phyiscotechnical Scientific Research Institute]

[Abstract] The great difference in the parameters of elementary cells of Ge and Si (4.2%) complicates heteroepitaxy of their solid solutions on Si because of a tendency to the formation and development of three-dimensional nucleation centers as the Ge concentration increases. For this reason, one of the main problems when growing heteroepitaxial layers of  $Ge_xSi_{1-x}$  from molecular flows in a vacuum is to improve the preparation of the Si surface;

this in turn facilitates the maximum reduction in the temperatures of preliminary annealing of the bases and the growth of the layers. The study reported herein examined the structure of heteroepitaxial layers of  $Ge_xSi_{1-x}$  and superlattices based on them that were produced on Si bases in a quasi-oilless vacuum of about  $2 \cdot 10^{-6}$  Pa. The molecular flow of Si was created by sublimating a small bar made of a high-ohm monocrystal, and the Ge was vaporized from a crucible. Bases from Si with an orientation of (100) were subjected to careful chemical treatment, including neutralization, etching to a depth of about 20  $\mu m$ , and passivation of the surface by an oxide lay in an ammonium-peroxide solution. The bases were vacuum-annealed at 800°C for 5 minutes under conditions of a molecular flow of Si with an intensity of about  $1 \cdot 10^{14} \text{ cm}^{-2}\text{s}^{-1}$ , thus ensuring a of the base that made it possible to reduce the temperature of growing the heteroepitaxial layers to 650°C and the concentration of Ge to 20 atm%.  $Ge_xSi_{1-x}/Si$  consisting of alternating (according to a periodic law) layers of Si and layers of solid solution were thus grown. The superlattices' periods varied from 17 to 210 nm, with the total number of layers amounting to 80 and the concentration of Ge in the heteroepitaxial layers reaching 20 atm%. X-ray diffraction studies of the layers confirmed the elastically stressed state of the solid solution's layers. The intensity of the satellite reflections from the superlattices was found to be in good agreement with the "step" model of the profile of the change in the Ge concentration. Figure 1, table 1; references 6: 2 Russian, 4 Western.

UDC 546.681'19:539.216.2:537.226

### Thermal Oxidation of GaAs/ $Bi_2O_3$ Structures in Oxygen

917M0022C Moscow IZVESTIYA AKADEMII NAUK  
SSSR: SERIYA NEORGANICHESKIYE MATERIALY  
in Russian Vol 26 No 10, Oct 90 (manuscript received  
2 Nov 88) pp 2030-2032

[Article by I.Ya. Mittova, V.V. Sviridova, V.N. Semenov, M.P. Kaplya, and Ye.N. Nevryuyeva, Voronezh State University]

[Abstract] Interest in GaAs-based integrated circuits possessing high speed and increased heat and radiation resistance has increased in recent years, hence the importance of the problem of forming a reliable dielectric coating at relatively low temperatures and with very high rapid growth rate. The study reported herein examines the formation of dielectric films on GaAs by thermooxidation of GaAs/ $Bi_2O_3$  structures and the growth, make-up, and selected physicochemical properties of the oxide layers grown. Chemically and mechanically polished plates of grade SAGOCH-1 monocrystalline GaAs oriented in the plane (III) with a specific resistance of  $\rho = 0.0010$  to 0.018  $\Omega \text{cm}$  were used for the experiment. The surface of the GaAs was subjected to preoxidation treatment in concentrated hydrofluoric acid for 20 minutes and then rinsed in distilled water. Pure  $Bi_2O_3$  was used as the starting substance. The thermooxidation kinetics of the GaAs were studied on freshly prepared GaAs/ $Bi_2O_3$  structures; the applied layers were between 130 and 140 nm thick. The resultant structures were oxidized in a Izoprin-type quartz

reactor with resistive reheating. The working temperature was regulated within  $+- 2^\circ\text{C}$  in the interval from 350 to 500°C, and the time was varied from 1 to 60 minutes. The thickness of the applied and oxidized layers was determined by the ellipsometric method. A  $Bi_2O_3$  layer on the surface of a GaAs semiconductive compound was indeed found to change the superconductor's thermooxidation kinetics. Two stages of the process were discovered. In the first, the transit reaction of the impurity oxide and base is decisive, and the acceleration of the process (versus that of an "impurity-free" process) reaches its maximum (up to 8-fold depending on the thickness). The second stage entails further transformation of the film's make-up mainly owing to the diffusion of the base's components into it. Preliminary application of  $Bi_2O_3$  onto a GaAs surface makes it possible to produce rather thick layers in comparatively short amounts of time. The electric strength of the specified films is, for practical purposes, no different from that for "pure" oxides on GaAs. Figures 2; references 6: 5 Russian, 1 Western.

UDC 539.216.2:546.48'47'22

### Properties of Thin Films of Solid Solutions of $Cd_xZn_{1-x}S$

917M0022D Moscow IZVESTIYA AKADEMII NAUK  
SSSR: SERIYA NEORGANICHESKIYE MATERIALY  
in Russian Vol 26 No 10, Oct 90 (manuscript received  
12 Oct 88) pp 2030-2032

[Article by V.N. Semenov, Ye.M. Averbakh, and Ya.A. Ugay, Voronezh State University imeni Leninskiy Komsomol]

[Abstract] Solid solutions of  $Cd_xZn_{1-x}S$  have different forbidden zone widths ( $\Delta E_{Cds} = 2.4$  and  $\Delta E_{ZnS} = 3.6$  eV). This fact makes it possible to vary their properties within broad bounds. Although a great deal of research has been conducted on bulk and powder CdS-ZnS specimens, little information exists on CdS-ZnS in the form of thin films. The study reported herein examined the electrical, optical, photoelectric, and luminescence properties of thin  $Cd_xZn_{1-x}S$  films obtained by the method of spraying solutions onto a heated base. The study also examined the link between layers' properties and their compositions. Films were produced on pyroceram and quartz bases in the temperature range from 550 to 600 K. Distilled water, ultrapure thiourea, and analysis-grade  $ZnCl_2$  and  $CdCl_2$  were used as the starting components. The concentration of salt solutions amounted to 0.1 mol/l, and that of the  $N_2H_4CS$  solution was 0.2 mol/l. The required pH level (2.5) was achieved by adding hydrochloric acid. A continuous series of solid  $Cd_xZn_{1-x}S$  solutions was synthesized; the average crystallite diameter was 250 nm. A compensation circuit using a VK 2-16 electrometer was used to study the films' electric and photoelectric properties. An SF-4 monochromator and VSU 2-P spectrophotometer were also used in the studies. The composition dependence of the width of the forbidden zone of thin  $Cd_xZn_{1-x}S$  solid solutions manifests only a slight deviation from nonlinearity. At 300 K, a minimum of the curve representing the concentration dependence of electric conduction is observed close to the

composition  $Cd_{0.25}Zn_{0.75}S$ . The concentration dependences of the photoelectric and luminescence parameters are governed by general laws that are characteristic for discontinuous solid solutions as well as by a number of anomalies arising on account of the distinctive features of the thin-film state and structural defects that arise under the given experiment conditions. Figures 2; references 11: 10 Russian, 1 Western.

UDC 541.136.2

### Electrochemical Properties of Titanium and Niobium Oxides-Fluorides

917M0022E Moscow *IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY* in Russian Vol 26 No 10, Oct 90 (manuscript received 17 Nov 88) pp 2168-2171

[Article by A.T. Falkengof, Ye.V. Makhonina, B.M. Zhigarnovskiy, V.S. Pervov, and V.A. Bogdanovskaya, General and Inorganic Chemistry Institute imeni N.S. Kurnakov, USSR Academy of Sciences, and Electrochemistry Institute imeni A.N. Frumkin, USSR Academy of Sciences]

[Abstract] Compounds with the structure  $ReO_3$  are currently enjoying wide-scale use because they may be used as current sources with a lithium anode. Titanium and niobium oxide-fluorides have a structure that is similar to the aforementioned compounds. The literature includes data regarding the possibility of deep intercalation of  $TiOF_2$  and  $NbO_2F$  by n-butyl lithium. These data have spawned the conclusion that oxide-fluorides are promising as anode materials for chemical current sources with a lithium anode. The study reported herein examined the possibility of the electrochemical intercalation of titanium and niobium oxide-fluorides.  $TiOF_2$  and  $NbO_2F$  were produced by dissolving the respective metal oxides in concentrated hydrofluoric acid and subsequently evaporating the solution. A mixed oxide-fluoride with the make-up  $Ti_{0.95}Nb_{0.05}O_{1.05}F_{1.95}$  was obtained in an analogous manner by dissolving calculated quantities of  $TiOF_2$  and  $Nb_2O_5$ . The individuality of the compounds was demonstrated by x-ray phase analysis. Analysis-grade  $LiClO_4$  was purified by recrystallization and then vacuum-dried at 160°C for several days. The purified, vacuum-distilled propylene carbonate used contained less than 1·10<sup>-2</sup>% water. The electrochemical studies were conducted in a dry box in an argon atmosphere in a cell with an anode and cathode that were separated by space. A PI-50-1 potentiostat and PR-8 programmer were used to make the measurements, lithium foil was used as an anode, and the standard electrode was produced by pressing metallic lithium into a glass tube with a lead. The cathode mixture, which consisted of 74% of the test substance, 13% acetylene black, and 13% powder polyethylene, was pressed into a nickel grid at 150°C and a pressure of 10<sup>7</sup> Pa. An S8-17 oscilloscope was used to record the chronopotentiometric

curves, and a DRON-0.5 diffractometer was used to perform the x-ray phase analysis. It was discovered that titanium and niobium oxide-fluorides may indeed be electrochemically intercalated by lithium; however, the rate of intercalation is low on account of the low value of the coefficient of lithium diffusion. Electrochemical deintercalation is difficult because of the interaction of lithium with the crystalline lattice of the oxide-fluorides studied. Figures 2; references 9: 1 Russian, 8 Western.

UDC 54-165

### The Production and Physical Properties of Crystals of $Cd_xZn_yHg_{1-x-y}Te$ Solid Solutions

917M0022F Moscow *IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY* in Russian Vol 26 No 10, Oct 90 (manuscript received) pp 2219-2220

[Article by I.S. Virt, D.I. Tsutyura, and D.D. Shuptar, Drogobych Pedagogical Institute imeni I. Franko]

[Abstract]  $Cd_xZn_yHg_{1-x-y}Te$  solid solutions are among the most intensely studied semiconductor materials. Because the width of their forbidden zone can be smoothly varied and because of their high charge carrier mobility and photosensitivity (close to theoretical), they are promising for use in creating instruments for use in infrared photoelectronics. Along with their advantages, however, they also possess a number of drawbacks, specifically, a high sensitivity to temperature and mechanical effects. These are due to the weak bond of the mercury atoms in the crystalline lattice, which in turn results in a low threshold of the generation of the semiconductors' inherent defects and is one of the reasons why instruments based on  $Cd_xZn_yHg_{1-x-y}Te$  are subject to accelerated degradation during their operation. Consequently, a method of increasing the threshold at which these semiconductors' inherent defects are generated must be found. The study reported herein examines the main physical properties of quaternary solid solutions of  $Cd_xZn_yHg_{1-x-y}Te$  produced by the Bridgman-Stockbauer method. The crystals were produced in thick-walled quartz ampoules. The following served as starting materials (atm%): Te, 50; Hg, 36; Cd, 1.25; and Zn, 1.5. The ampoules with specimen were held at a temperature of 30°C above the liquidus point for 50 hours with continuous stirring of the mixture and were then released into a furnace at a rate of about 1 mm/h. The temperature gradient in the furnace amounted to about 50°C/cm. To homogenize the crystal, the ampoules were annealed at 550°C for 250 hours. Significant segregation of the components was observed in the resultant ingots. It was found that adding zinc increases the stability of photosensitivity by a factor of about 1.5. The increased microhardness observed also provides a basis for concluding that the material develops an increased resistance to mechanical effects. Thus,  $Cd_xZn_yHg_{1-x-y}Te$  crystals are found to possess more stable parameters and greater strength than do  $Cd_xHg_{1-x}Te$  crystals. Figures 2; references 2: 1 Russian, 1 Western.

UDC 546.431;546.16;546.215

**Synthesis, Properties, and Kinetics of Thermal Decomposition of the Peroxosolvate of Barium Fluoride  $\text{BaF}_2 \cdot 2\text{H}_2\text{O}_2$**

917M0025 Moscow ZHURNAL  
NEORGANICHESKOY KHMII in Russian Vol 35  
No 10, Oct 90 (manuscript received 10 Apr 90)  
pp 2483-2487

[Article by K.V. Titova, Ye.I. Kolmakova, and V.Ya. Rosolovskiy, Institute of New Chemical Problems, USSR Academy of Sciences]

[Abstract] Fluorides of alkali metals are well soluble in hydrogen peroxide and form the peroxosolvates  $\text{KF}\text{H}_2\text{O}_2$ ,  $\text{KF}2\text{H}_2\text{O}_2$ ,  $\text{KF}3\text{H}_2\text{O}_2$ ,  $\text{RbF}1.5\text{H}_2\text{O}_2$ , and  $\text{CsF}\text{H}_2\text{O}_2$ . The solvates are easily crystallized from fluoride solutions in anhydrous hydrogen peroxide or from aqueous solutions. Far less is known about the reaction of hydrogen peroxide with fluorides of alkali earth metals. All that is known is that fluorides of alkali earth metals are sparingly soluble in both hydrogen peroxide and water and that the solvate only forms barium fluoride. To gain more knowledge in this area, the authors of the study reported herein examined the reaction of barium fluoride with hydrogen peroxide in the  $\text{H}_2\text{O}_2$  concentration range from 10 to 100%. Analysis-grade barium fluoride was reacted with different concentrations of hydrogen peroxide that were produced by holding a 30%  $\text{H}_2\text{O}_2$  solution over sulfuric acid in a desiccator. Permanganometric titration was used to analyze the  $\text{H}_2\text{O}_2$  solutions and solid reaction products for their  $\text{H}_2\text{O}_2$  content. Infrared spectra were taken on a UR-20 spectrometer, and thermograms were recorded on an NTR-64. The furnace was heated at a rate of 6-7°/min, and  $\text{Al}_2\text{O}_3$  was used as a standard. Specimens of

$\text{BaF}_2 \cdot 2\text{H}_2\text{O}_2$  obtained under identical conditions (at a hydrogen peroxide concentration of 50 mass percent and with an  $\text{H}_2\text{O}_2:\text{BaF}_2$  ratio of 1:5 for 8 hours) were used to extract the kinetic curves. The reaction kinetics were studied in the temperature range from 85 to 105°C. The degree of decay was determined as the ratio of the volume of oxygen released up to the given moment in time to the total volume of oxygen corresponding to the total decomposition of hydrogen peroxide contained in the weighted portion. It was found that the process of solvation of barium fluoride by hydrogen peroxide occurs not only in a medium of anhydrous hydrogen peroxide but also in its aqueous solutions. Extraction of hydrogen peroxide by barium fluoride occurs at hydrogen peroxide concentrations of as little as 10 mass percent. The solvation reaction was found to occur rather rapidly; within 2 hours, there was quantitative formation of peroxosolvate. Solvate with the threshold composition formed when the equilibrium hydrogen peroxide concentration was at least 20 mass percent (below this percentage solvation was incomplete). The barium fluoride peroxosolvate obtained from aqueous hydrogen peroxide solutions was a white, highly dispersed, little-hygrosopic powder, whereas the  $\text{BaF}_2 \cdot 2\text{H}_2\text{O}_2$  obtained from anhydrous hydrogen peroxide was less disperse. Under conditions of continuous heating, the solvate  $\text{BaF}_2 \cdot 2\text{H}_2\text{O}_2$  decomposes intensively at 130-140°C with a significant exothermal effect. The kinetics of the thermal decomposition of barium fluoride peroxosolvate is S-shaped in the temperature range from 85 to 105°C. A significant slowdown is observed in the later stages. Even though  $\text{BaF}_2 \cdot 2\text{H}_2\text{O}_2$  is less stable than  $\text{KF}\text{H}_2\text{O}_2$ , using the form as a solid carrier of hydrogen peroxide is still entirely possible. It can be used to obtain virtually salt-free hydrogen peroxide solutions, which is important since such solid carriers of hydrogen peroxide have not existed previously. Figures 2, tables 2; references 6: 5 Russian, 1 Western.

UDC 546.123.2+548.55

**Optical and Electromigration Properties of Thallium Chalcogermanates (Stannates)**

917M0022G Moscow *IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY* in Russian Vol 26 No 10, Oct 90 (manuscript received 11 Jul 88) pp 2047-2049

[Article by V.F. Zinchenko, A.V. Shapovalov, G.A. Teterin, Yu.V. Vorobyev, and I.S. Chaus, Physicochemical Institute imeni A.V. Bogatskiy, UkrSSR Academy of Sciences]

[Abstract] Thallium chalcogermanate- and thallium chalcostannate-type compounds are beginning to enjoy wide-scale use in the manufacture of photosensitive elements for semiconductor instruments. Previous studies of the phase diagrams of the systems  $Tl_2X-GeX_2$  and  $Tl_2X-SnX_2$  (where  $X = S$  and  $Se$ ) have established the formation of the compounds  $Tl_4Ge(Sn)X_4$ ,  $Tl_2Ge(Sn)X$ , and  $Tl_2Ge_2(Sn_2)X_5$ , and their electrical (specific resistance at room temperature) and optical properties have been rather well systematized. More research on the infrared transmission spectra of the aforementioned materials is needed, however. Complicating the problem further is the fact that the data published on the nature of the melting of thallium dithiogermanate and on the width of the glass formation region in  $Tl_2X-GeX_2$  systems are contradictory. The study reported herein represents an attempt to refine these data by studying the temperature dependence of the electroconduction of thallium thio- and selenogermanates in the liquid state and during transition through the crystallization point. The complex chalcogenides used were prepared by direct synthesis from the elements in accordance with the method described elsewhere. A Perkin-Elmer 580 spectrophotometer was used to take the IR-range absorption spectra of the polycrystalline chalcogenide materials all the way to  $200\text{ cm}^{-1}$  by dispersing the matter in vaseline oil. The concentration of dispersed particles and spectrum-recording modes used were selected empirically for each material. The authors established a link between the position of the first peak of absorption in the IR range and the structurokinetic characteristics of the  $Ge(Sn)-X$  bonds. It was also found that the electroconduction of  $Tl_2GeS_3$  and  $Tl_2GeSe_3$  melts decreases abruptly at the crystallization point and that the nature of its temperature dependence changes. The electroconduction of the compounds  $Tl_2Ge_2S_5$  and  $Tl_2Ge_2Se$  remains practically constant during melting, which indicates their pronounced tendency toward glass formation. Figures 3; references 11: 10 Russian, 1 Western.

UDC 539.213.23

**The Search for Ways of Stabilizing the Amorphous Structure of Electrophotographic Selenium Layers**

917M0022H Moscow *IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY* in Russian Vol 26 No 10, Oct 90 (manuscript received 10 Oct 88) pp 2097-2099

[Article by V.O. Shvalev, Kurgan State Pedagogical Institute]

[Abstract] A large number of works have studied the structure and crystallization of amorphous selenium layers; however, the kinetic aspects of the process of crystallization of amorphous selenium layers of crystalline and amorphous bases have been little studied even though the topic is important from both scientific and practical standpoints. This article uses electron and light microscopy and electrical resistance methods to study the kinetics of the crystallization of amorphous selenium layers that have been produced by vacuum precipitation from a molecular beam onto crystalline ( $SnO_2$  and mica) and noncrystalline (glass, carbon, and chalcogenide glasses with the make-up  $As_xSe_{1-x}$ ) bases. The nucleation process of all of the bases examined was found to be heterogeneous in nature. Layers of selenium deposited onto bases of  $As_xSe_{1-x}$  glasses were found to have the lowest crystallizability and, consequently, the most stable amorphous state. Compared with films of the same thickness precipitated onto other bases, selenium films on bases of  $As_xSe_{1-x}$  had the maximum values of latent periods of natural and artificial aging and periods of complete mass crystallization and nucleation and the minimum spherolite growth rates and minimum integrated degree of crystallizability under identical conditions of isothermal annealing. Thus, from the standpoint of amorphous stability of the state of condensed selenium films, those bases of  $As_xSe_{1-x}$  chalcogenide glasses thus turned out to be the optimal among those examined. A method of stabilizing the amorphous state of vacuum condensates of selenium was proposed. It entails transforming the "sharp" interface between bases made of  $As_xSe_{1-x}$  and selenium films into an intermediate layer ( $As_xSe_{1-x} + Se$ ) with a smooth reduction in the arsenic concentration. Table 1; references 16: 10 Russian, 6 Western.

UDC 666.3

**Reaction of  $YBa_2Cu_3O_{7-x}$  Ceramic With Oxygen and Carbon Dioxide**

917M0022I Moscow *IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY* in Russian Vol 26 No 10, Oct 90 (manuscript received 11 Nov 88) pp 2181-2183

[Article by A.A. Budanov, O.D. Torbova, Ye.N. Kurkin, V.N. Troitskiy, V.V. Ganin, and A.V. Buluyev, New Chemical Problems Institute, USSR Academy of Sciences]

[Abstract] It is widely known that it is the oxygen content in a  $YBa_2Cu_3O_{7-x}$  compound that determines that compound's superconductivity. In addition, the critical superconductivity parameters of polycrystalline specimens depend on their microstructure. It is particularly dependent on the presence of impurities in the grain boundaries of the superconductive phase. Carbon is one of the most likely impurities in  $YBa_2Cu_3O_{7-x}$  ceramic because barium carbonate is often used to synthesize superconductive compounds and the temperature of its complete decomposition at atmospheric pressure exceeds the synthesis temperature. The presence of an alkali-earth element in a complex oxide gives it the properties of a base and, consequently, the possibility of absorbing  $CO_2$  when the

material makes contact with air. That is probably one of the reasons for the degradation of superconductivity when specimens of high-temperature superconductors are stored in air. The study reported herein used thermographic, mass spectral, and roentgeographic analysis methods to study the reaction of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  ceramic with oxygen and carbon dioxide in the temperature range from 20 to 950°C. The authors managed to establish the phase transition of an orthorhombic modification of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  into a tetragonal modification with the following elementary cell parameters (nm):  $a = 0.3887$  and  $c = 1.1897$  at 650°C in an oxygen atmosphere. Above 400°C, the oxygen content in the compound is determined by the mobile equilibrium between the gas and condensed phases. When the specimens absorbed carbon dioxide, carbonates were formed along the grain boundaries of the superconductive phase. These prevented oxygen saturation during subsequent heat treatment and reduced the critical current density. Figures 3; references 2: 1 Russian, 1 Western.

UDC 537.228.1+66.046

### Sintering Niobate Piezoceramic From Finely Dispersed Powders

917M0022J Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 10, Oct 90 (manuscript received) pp 2190-2193

[Article by L.A. Reznichenko, T.V. Donskova, O.N. Razumovskaya, V.D. Komarov, L.S. Ivanova, L.A. Shilkina, and V.A. Aleshin, Rostov State University and Physics Research Institute]

[Abstract] Because piezo materials must have a high density, high uniformity, and highly reproducible properties, finely dispersed raw material with a particle size of  $<1\text{ }\mu\text{m}$  must be used when synthesizing them. The same applies for the niobium pentoxide that is the main raw material for niobate piezoceramic based on niobates of alkaline metals. Methods have recently been developed for producing finely dispersed ( $D < 1\text{ }\mu\text{m}$ ) niobium pentoxide with a purity that, albeit high, is much below the grading ultrapure, which permits the manufacture of products with high piezo characteristics. Achieving such materials requires knowing the optimal dispersion of niobium pentoxide, i.e., that dispersion that would provide the best-quality niobate piezoceramic. In an effort to gain this knowledge, the authors of this article conducted a comprehensive study of the properties of solid solutions based on sodium-lithium niobates that were synthesized from finely dispersed  $\text{Nb}_2\text{O}_5$  produced by the heterophase method. Different dispersions were achieved by varying the duration for which low-water niobium hydroxide (an amorphous product of the heterophase interaction of  $\text{NbCl}_5$  with a 25% solution of  $\text{NH}_4\text{OH}$ ) was dried at 670 K from 1.2 to  $3.6 \cdot 10^3$  seconds and by then crystallizing it at 840 and 1,170 K. The change in the parameters of the microstructure, crystalline structure, and electrophysical properties of the piezoceramic as a function of the dispersion of the niobium pentoxide produced by the heterophase method was found to be nonmonotonic in nature. The ceramic's average grain size and uniformity of the grains' size

distribution were determined to be due to the distinctive features of the recrystallization process. The formation of coarse-grained, uniformly sized grains of ceramic made of powders with a specific surface between 2,500 and 3,500  $\text{m}^2/\text{kg}$  was found to facilitate the realization of a crystalline structure with a large number of domain boundaries and with increased mobility. It was demonstrated that piezoceramic manufactured from  $\text{Nb}_2\text{O}_5$  with a specific surface area of about 3,000  $\text{m}^2/\text{kg}$  has the optimal properties. Figure 1; references 13 (Russian). I

UDC 541.64:539.2

### Production and Properties of Polyphosphate Soldered Joints on Ceramic

917M0022K Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 10, Oct 90 (manuscript received 12 Oct 88) pp 2198-2201

[Article by G.G. Khisameyev, O.S. Sirokin, and R.S. Sayfullin, Kazan Chemical Technology Institute imeni S.M. Kirov]

[Abstract] "Ceramic-soldered joint-ceramic" composites may be created during gas-phase reactions. A polyphosphate soldered joint produced in such a manner may be used to bind solid composites or as a unique type of filter. The study reported herein examined the distinctive features of the production of polyphosphate soldered joints, their structures, and some of their properties. Gas-phase synthesis was performed on a unit described elsewhere. Gas-phase  $\text{P}_4\text{O}_{10}$ ,  $\text{P}_4\text{O}_{10}\text{-H}_2\text{O}$ , and  $\text{P}_4\text{O}_{10}\text{-H}_2\text{O-HF}$  were produced by respectively evaporating phosphorus oxide, an 85% solution of monophosphoric acid, and a 1:2 mixture of monophosphoric acid with a 40% solution of hydrofluoric acid. In all of the experiments, the evaporation temperature (i.e., the temperature at which the gas-phase mixture was synthesized) corresponded to the temperature of producing the soldered joint. The following types of rods with a contact area of  $60\text{ mm}^2$  were used in the experiments: steatite ceramic rods containing  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{BaO}$ ,  $\text{MgO}$ , and  $\text{SiO}_2$  and aluminum oxide ceramic (VK-94-1) rods containing  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{MnO}_2$ , and  $\text{Cr}_2\text{O}_3$ . The joints were produced by directly treating joined specimens in a gaseous mixture or by heating the specimens with preapplication of a layer of polyphosphates on their ends. In the case of the first technique, the rods were heated to a temperature of 400-1,200°C and then treated in the gas mixture for 10 minutes. In the case of the second technique, the materials being joined were first subjected to treatment in a gas mixture for 10 minutes at 1,000°C and were then joined at the ends and heated for 10 minutes at 400-1,200°C in the absence of a gas-phase. No glassy joint formed in the gaseous  $\text{P}_4\text{O}_{10}$  or  $\text{P}_4\text{O}_{10}\text{-H}_2\text{O}$ . This was evidently due to the very narrow vitrification regions in the system  $\text{Al}_2\text{O}_3\text{-P}_4\text{O}_{10}\text{-SiO}_2$ . Producing a glassy soldered joint in the gaseous mixture required the addition of hydrogen fluoride that activated a reduction in the activation barrier of the reaction between the ceramic and gaseous mixture in accordance with a halogenation mechanism. The soldered joint obtained in the  $\text{P}_4\text{O}_{10}\text{-H}_2\text{O}$  gaseous mixture between steatite specimens at 400°C

under the effect of steam disintegrated within 10-15 minutes. The soldered joint produced in an anhydrous medium disintegrated even more quickly. This was due to the lesser degree of cross-linking of the polyphosphate chains by the cations diffused during gas-phase synthesis from the ceramic into the make-up of the melt when  $P_4O_{10}$  was used and to the increased degree of cross-linking when  $P_4O_{10}\cdot H_2O$  and  $P_4O_{10}\cdot H_2O\cdot HF$  gaseous mixtures were used on account of the formation of more reactive mixtures, i.e., ultraphosphoric acids. Increasing the temperature at which the soldered joints were treated in  $P_4O_{10}\cdot H_2O\cdot HF$  resulted in an increase in the soldered joints' moisture resistance and microhardness. It was found that specimens could be joined by direct treatment in a gas-phase at a temperature of 400°C, whereas joining specimens by using the technique of preapplication of a layer of polyphosphates required a temperature of at least 750°C. Figures 3; references 15 (Russian).

UDC 546.28

### Change in the Physicochemical Properties of p-Silicon Under the Effect of High-Energy Electrons

917M0022L Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 10, Oct 90 (manuscript received 21 Mar 89) pp 2210-2211

[Article by B.R. Meshcherov and V.I. Tumanov, Atomic Energy Institute imeni I.V. Kurchatov, USSR Academy of Sciences]

[Abstract] While working on developing a method for layer-by-layer measurement of activity in an experiment studying the passage of ultrarelativistic electrons through monocrystals, the authors of this concise communication irradiated a silicon target 370  $\mu m$  thick that had an initial resistance of about 1  $\Omega\cdot cm$  (with a boron atom concentration of about  $2\cdot 10^{16} cm^{-3}$ ). The irradiation was generated on a pulse accelerater at an electron energy of about 50 MeV and a mean current of about 150  $\mu A$ . The total electron dose amounted to about  $10^{18} cm^{-2}$ . The electron beam on the crystal had a cross sectional area of about 10  $cm^2$ , whereas the total area of the target amounted to about 30  $cm^2$ . The silicon plate was mounted in air behind the exit window of an electron guide; according to calculations, its temperature should have ranged from 400 to 450°C. After the irradiation, the crystal was subjected to repeated cycles of chemical etching and film application. The irradiated and nonirradiated portions of the crystal were compared from the standpoint of a variety of parameters, including thickness and electric conduction. On the basis of their research, the authors conclude that earlier research discounting the effect of the type or magnitude of the conduction of silicon on the thickness of an anodic oxide film grown in a constant current is in fact imprecise. The authors of the present study go on to assert that their own research confirms the marked role of electronic current in the process of anodizing silicon. They feel that it is entirely possible that a study of the growth of an anode oxide film on specimens with a low concentration of free carriers will make it possible to establish the mechanism of

the effect of conduction electrons on the ionic current. They cite previous research in their explanation of the reduction in the rate of chemical etching in the irradiated region of the crystal. References 8: 5 Russian, 3 Western.

UDC 541.183

### Using the X-Ray Photoelectron Spectroscopy Method To Study a Gallium Arsenide Surface

917M0022M Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 10, Oct 90 (manuscript received 11 Nov 87) pp 2213-2215

[Article by I.A. Kirovskaya, V.G. Shtabnov, A.V. Pashis, and Ye.D. Skutin, Omsk Polytechnic Institute]

[Abstract] The general characteristics of the chemical and charge state of an actual gallium arsenide surface have been established on the basis of previously conducted thermal desorption, mass spectrometric, optical, magnetic, electrophysical, and other types of research. The study reported herein used the method of x-ray photoelectron spectroscopy to complement and expand this research. A VGESCA-3 with  $AlK_{\alpha}$  radiation ( $E = 1,486.6$  eV) was used to record the x-ray photoelectron spectra of monocrystalline gallium arsenide wafers [orientation, (100)] with the dimensions  $0.8 \times 6 \times 8$ . They obtained data on the electron bond energy, atomic ratios, and values proportional to the percent content of elements of gallium arsenide specimens treated in the following ways: protracted storage in air; oxidation in air (temperature, 1,070 K; duration, 15 minutes); treatment with etching agent (9% KOH + 30%  $H_2O_2$ ); oxidation in oxygen (temperature, 773 K; duration, 45 minutes); irradiation with argon ions (energy, 10 keV); and holding in air for 10 minutes after bombardment. These data are summarized in table form. Table 1; references 4 (Russian).

UDC 621.315.592.2:546.681'19

### Effect of Transient Processes in the Gaseous Phase on the Properties of Semiconductor Structures Produced by Organometallic Compound-Hydride Epitaxy

917M0022N Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 10, Oct 90 (manuscript received 4 Nov 86) pp 2215-2217

[Article by P.B. Boldyrevskiy and O.I. Khrykin]

[Abstract] This article presents the results of research on transient processes occurring in a gas phase as its make-up is controlled. Also examined are the effects of these processes on the properties of multilayer homo- and heteroepitaxial structures grown in a system using organometallic compounds and hydrides. Specifically, the authors examine the process of producing layers of gallium arsenide that have been  $\delta$ -doped with germanium. The authors present equations for the following: the duration of concentration transitions in a multilayer epitaxial structure in the case of a staged change in concentration or

switching of the components of the gas mixture at the entry to the reaction chamber; the flow of germanium impurity atoms onto a gallium arsenide surface; the growth rate of  $_{1-x}Al_xAs$  layers (i.e., growth by the method of organometallic compound-hydride epitaxy); and the length of a heterojunction. During the course of this concise report, the authors show the effect of transient processes occurring in a gas phase on the formation of semiconductor homo- and heterostructures with sharp transitions between the layers by the method of organometallic compound-hydride epitaxy. They establish that the main factors determining the extent of junctions are the growth rate of the layers and its nonstationarity in the case of epitaxy of  $_{1-x}Al_xAs$  as well as the inertia of the system when the necessary concentrations of the gas-phase mixture's components are established. Figures 2; references 6: 4 Russian, 2 Western.

UDC 546.791'24:538.21

### Magnetic Properties of USbBi

917M00220 Moscow IZVESTIYA AKADEMII NAUK SSSR: SERIYA NEORGANICHESKIYE MATERIALY in Russian Vol 26 No 10, Oct 90 (manuscript received 26 Oct 88) p 2222

[Article by V.I. Chechernikov, L.F. Martynova, N.T. Kuznetsov, V.K. Slovyanskikh, and N.V. Gracheva, Institute of General and Inorganic Chemistry imeni N.S. Kurnakov, USSR Academy of Sciences]

[Abstract] The compounds  $USb_2$  and  $UBi_2$  are antiferromagnetics with a Neel temperature ( $T_N$ ) of 206 and 183, respectively. The study summarized in this concise communication examined their magnetic properties. The test specimens were obtained by the vacuum-thermal method from elements in graphitized quartz ampules at 1,200 K for 5 hours. The specimens were then subjected to differential thermal analysis in hermetically sealed molybdenum crucibles. The compounds  $USb_2$  and  $UBi_2$  formed at temperatures of 1,630 and 1,285 K. The trinary compound  $USbBi$  fuses congruently at 1,620 K. X-ray phase and microstructural analysis showed that after annealing both specimens were single-phase specimens.  $USbBi$  is isostructural with  $USb_2$  and  $UBi_2$  and crystallizes in a tetragonal structure. The parameters of the elementary cell are  $a = b = 0.428$  and  $c = 0.872$  nm. In the range from 80 to 170 K, the magnetic susceptibility of  $USbBi$  is virtually independent of temperature. At higher temperatures there is a change in magnetic susceptibility that is characteristic for antiferromagnetics with a fuzzy maximum, i.e.,  $T_N = 230$  K. Replacing the bismuth atoms by surmium atoms did not change the type of magnetic order; rather, it only increased the Neel temperature by several degrees. Figure 1; references 3: 2 Russian, 1 Western.

UDC 541.1

### Nondestructive Testing of the Percent Content of the Superconductive Phase in High-Temperature Semiconductor Synthesis Products

917M0025A Moscow ZHURNAL NEORGANICHESKOY KHIMII in Russian Vol 35 No 10, Oct 90 (manuscript received 20 Dec 89) pp 2451-2455

[Article by Ye.A. Volkov, A.V. Filatov, and V.M. Novotortsev]

[Abstract] One of the main tasks arising during the synthesis of high-temperature superconductors [HTSC] is that of determining the percent content of superconductive phase in the specimens produced. In view of the fact that magnetism is an inextricable property of the superconductive state and in view of the relative simplicity and universality of magnetic measurements, the authors of the study reported herein sought to develop a method of accomplishing the aforementioned task by examining the link between the moment of a frozen-in flow and the magnetization of superconductive specimens containing a superconductive phase in specimens with the make-ups  $Y-Ba-Cu-O$ ,  $Bi-Sr-Ca-Cu-O$ , and  $Tl-Ba-Ca-Cu-O$  in the temperature range from 77 to 117 K. They used the data obtained during their experiments as the basis for proposing a nondestructive method of determining the percent content of superconductive phase in HTSC ceramic with strongly bound superconductive granules screening the specimen's superconductive bulk. The method developed entails measuring the slope of the limiting return magnetization curve of the specimen in fields less than the first critical magnetic field. Measurements are taken for three mutually perpendicular orientations of the specimen so as to allow for the unknown demagnetizing factor of the superconductive granules. Superconductors with the make-ups  $Y-Ba-Cu-O$  and  $Tl-Ba-Ca-Cu-O$  were compared with respect to a number of parameters. These data were checked by pulverizing the ceramic specimens and then taking comparative measurements of the resultant powders. The authors recommend their proposed method as being simple and quick and affording the capability of making measurements without destroying the specimens undergoing testing. Figures 6, table 1; references 6: 2 Russian, 4 Western.

UDC 548.7;548.314

### Effect of the Distinctive Features of the Distribution of Oxygen Atoms in the Cu1 Plane on the Charge Carrier Distribution in $YBa_2Cu_3O_{6+x}$

917M0025B Moscow ZHURNAL NEORGANICHESKOY KHIMII in Russian Vol 35 No 10, Oct 90 (manuscript received 19 Apr 90) pp 2456-2458

[Article by V.P. Nifontov, V.I. Ponomarev, and L.O. Atovmyan, Chemical Physics Institute, USSR Academy of Sciences]

[Abstract] Experiments on the Hall effect have shown that for compounds in the series  $YBa_2Cu_3O_{6+x}$  to be superconductive it is necessary that the concentration of charge carriers (holes) in them be greater than some minimum value. Research has also established that as the concentration of holes increases, the temperature of the transition to the superconductive state also increases. It is further known that in the case of some rhombic and tetragonal compounds with identical values of  $x$  ( $x = 0.55$ ), only the rhombic one will make the transition to a superconductive state. It may be hypothesized that at equal values of  $x$ , a tetragonal structure will contain a lesser concentration of holes than a rhombic structure will and that, consequently,

the rhombic structure will not reach the concentration required for the manifestation of superconductivity. Through detailed sample calculations, the authors of this article demonstrate that the concentration of holes in  $\text{YBa}_2\text{Cu}_3\text{O}_{6+x}$  depends not only on  $x$  but also on the distinctive features of the distribution of oxygen atoms with respect to positions 04 and 05. They further establish experimentally that according to x-ray data some tetragonal crystals do indeed possess superconductive properties. This is explained by the fact that they consist of microregions that each contain oxygen atoms that are ordered in the plane Cu1 in a small chain and by the fact that the dimensions and orientation of these regions are such that the crystal looks tetragonal on the basis of x-ray data. In fact, there may be far fewer 04-05 short contacts and, thus, more holes. Figures 3; references 6: 1 Russian, 5 Western.

UDC 541.123.6:546.131:549,76

**Models of the Phase Equilibria of the Four-Component System  
(KF)<sub>2</sub>-(KCl)<sub>2</sub>-K<sub>2</sub>CO<sub>3</sub>-K<sub>2</sub>WO<sub>4</sub>**

917M0025C Moscow ZHURNAL

NEORGANICHESKOY KHMII in Russian Vol 35  
No 10, Oct 90 (manuscript received 20 Feb 90)  
pp 2652-2659

[Article by Zh.A. Koshkarov, A.M. Gasanaliyev, and A.S. Trunin, Karbadino-Balkarsk State University and Dagestanskiy Pedagogical Institute]

[Abstract] This article is ostensibly the first to use methods of differential and projective geometry to study the four-component system (KF)<sub>2</sub>-(KCl)<sub>2</sub>-K<sub>2</sub>CO<sub>3</sub>-K<sub>2</sub>WO<sub>4</sub>, which is a key component of the more complex six-component system Na, K/F, Cl, CO<sub>3</sub>, MoO<sub>4</sub>, WO<sub>4</sub>, which is in turn interesting from the standpoint of producing anticorrosion carbide coatings and heat-accumulating materials from melts. On the basis of their own previously developed method, the authors postulate a priori the crystallization tree of the system (KF)<sub>2</sub>-(KCl)<sub>2</sub>-K<sub>2</sub>CO<sub>3</sub>-K<sub>2</sub>WO<sub>4</sub>. They show that the tetrahedrizing cross section (KCl)<sub>2</sub>-K<sub>2</sub>CO<sub>3</sub>-K<sub>3</sub>FWO<sub>4</sub> divides this system into two stable subsystems, namely, (KCl)<sub>2</sub>-K<sub>2</sub>CO<sub>3</sub>-K<sub>3</sub>FWO<sub>4</sub>-K<sub>2</sub>WO<sub>4</sub> and (KCl)<sub>2</sub>-K<sub>2</sub>CO<sub>3</sub>-K<sub>3</sub>FWO<sub>4</sub>-(KF)<sub>2</sub>. First- and second-order analytic models of the combined crystallization of the two phases are used to calculate the coordinates of the quaternary noninvariant points. To confirm their crystallization tree and refine their calculated data, the authors conducted an experimental study of the four-component system (KF)<sub>2</sub>-(KCl)<sub>2</sub>-K<sub>2</sub>CO<sub>3</sub>-K<sub>2</sub>WO<sub>4</sub> by combining differential thermal analysis and the thermographic-projection method. The calculated data regarding the coordinates of the quaternary noninvariant points were found to be in good agreement with the experimental values. The authors conclude that their algorithm is the most effective algorithm available for formulation of a crystallization tree, analytic description of all of the phase equilibrium and calculation of the noninvariant points, and experimental confirmation and refinement by means of projective geometry. Figures 4, tables 2; references 18: 16 Russian, 2 Western.

UDC 546.33'845'185'171

**Production and Properties of  
Monoamidotrihydroxycyclotrinitridosulfodiphosphate  
Anion**

917M0025E Moscow ZHURNAL  
NEORGANICHESKOY KHMII in Russian Vol 35  
No 10, Oct 90 (manuscript received 5 Jan 90)  
pp 2492-2496

[Article by I.A. Rozanov, L.Ya. Medvedeva, and L.V. Goyeva, Institute of General and Inorganic Chemistry, USSR Academy of Sciences]

[Abstract] The research material that has been accumulated with regard to the synthesis and properties of compounds with a sulfur-phosphorus-nitrogen-containing cyclic anode, i.e.,  $[P_2N_3SO_2(NH_2)_4]^-$ , has made it possible to formulate a number of generalizations about their stability. One aspect of such compounds that has not been researched, however, is this anion's behavior in neutral and alkaline solutions when heated. In an attempt to fill this knowledge gap, the authors of the study reported herein examined the hydrolysis of the heterocyclic anion  $[P_2N_3SO_2(NH_2)_4]^-$  by way of the example of ammonium, sodium, and rubidium salts when acetates of the respective

cations were mixed in a water-and-dioxane solution. Analysis of the data from chemical, x-ray phase, and paper chromatographic analyses of the hydrolysis products indicated that hydrolysis of the anion does not affect the bonds of the cycle itself and that the skeleton of the new anion maintains the composition and structure of the initial phosphazene; rather, the process appears to occur along the exocyclic  $NH_2$  groups and to culminate in the stage of the substitution of three of the four  $NH_2$  groups by hydroxyl groups. A second distinction of the resultant anion is that although the hydrolysis of the anion was conducted in a medium with a sufficient excess of acetate salt of the univalent cation, no substitution of hydroxy group's hydrogen by cation was ever observed, i.e., the basicity of the new anion remained equal to 1 (like that of the initial anion). Analysis of the IR spectra of the compounds with a hydrolyzed anion confirms that at least one amido group is present in the salts since a full set of valence and deformation oscillations of the  $NH_4$  groups is observed in the spectra. The presence of P-OH groups in the salts with a hydrolyzed anion was also confirmed by the IR spectroscopy data. The type of reaction between cation and ligand and the state of the heterocycle in the hydrolyzed salts were generally maintained. Figure 1, tables 3; references 8: 7 Russian, 1 Western.

UDC 551.24+262.5+550.8

**Predicting Oil and Gas Catch Basin and Deposit Locations - Basis for Developing Raw Material Base**

907M0141A Kiev NEFTYANAYA I GASOVAYA  
*PROMYSHLENNOST* in Russian No 1, Jan-Mar 90  
 (manuscript received 21 Feb 89) pp 2-5

[Article by Ye. V. Zakharov and V. T. Tarakanov, IFING]

[Abstract] In view of the diminishing reserves of oil and gas in the older producing regions of the country, and the steadily growing fund of non-anticlinal folds discovered in the newer regions, the need for predicting hydrocarbon deposit locations consisting not only of the arched (anticlinal folds), but also lithological, stratigraphic, tectonic-screening, and other types, becomes apparent. In the present work some generalizations and analyses of geological and geophysical data are presented from which it is possible to determine dominant geological features for locating various oil and gas catch basins, primarily in the northwest shelf of the Black Sea. Figure 1; references 6 (Russian).

UDC 553.981:550.8(477.87)

**Prospecting Methods for Gas Deposits in Transcarpathia**

907M0141B Kiev NEFTYANAYA I GASOVAYA  
*PROMYSHLENNOST* in Russian, No 1, Jan-Mar 90  
 (manuscript received 29 Mar 89) pp 6-8

[Article by B. I. Denega, Yu. Z. Krupskiy, N. S. Predtechenskaya, Geological Prospecting SRI (Ukrainian)]

[Abstract] The Transcarpathian foredeep, while comprising 11.8 percent of the potential hydrocarbon resources of the western UkrSSR, has failed to produce any new deposits for a long period of time so that exploratory drilling has almost ceased entirely. Then in 1982 the first gas deposit in Transcarpathia (Solotvin) was discovered; then came the Russo-Komarov in 1985, and the Korolev in 1988. These deposits lie 2000-2200 meters deep in an economically developed area. An examination of the geological and gas-bearing features of the Russo-Komarov region shows that it is similar to the deposits in Eastern Slovakia, thereby permitting development of rational methods for conducting geological prospecting operations on analogous areas on the new Transcarpathian foredeep. Figure 1.

UDC 622.245.1

**Estimating Effectiveness Bore Hole Reinforcement Efforts**

907M0141D Kiev NEFTYANAYA I GASOVAYA  
*PROMYSHLENNOST* in Russian No 1, Jan-Mar 90  
 (manuscript received 16 May 88) pp 24-27

[Article by N. M. Soshnin, R. F. Ukhonov, and V. S. Fomichev, VNIIKR Petroleum]

[Abstract] One of the chief means for raising the longevity of oil wells and helping to conserve oil resources lies in the development and improvement of measures to increase the strength of bore holes. The latter relies on such factors as the composition and properties of tamping and buffer solutions, cementation technology, and devices used therewith. Frequently, an entire set of measures are undertaken, and the economic effect is figured on the basis of the entire complex and not on any single measure alone. The necessity for economic evaluation, especially during the design stage, is especially acute at the present time when funding of unprofitable operations has been curtailed. In the present work a new and more simplified statistical method is presented for determining the efficiency of individual bore holes. References 4 (Russian).

UDC 662.279.42

**Determination of Gas Displacement Efficiency During Cycling Process**

907M0141E Kiev NEFTYANAYA I GASOVAYA  
*PROMYSHLENNOST* in Russian No 1, Jan-Mar 90  
 (manuscript received 16 May 88) pp 29-31

[Article by V. S. Grigoryev and T. Ye. Prokofyeva, Ukrainian Gas SRI]

[Abstract] A basic problem in designing a cycling process for heterogeneous gas-condensate deposits lies in predicting the dynamics of the capture coefficient of displaced formation gas as a function of the volume of gas pumped for a given distribution of operating and injection wells in a formation. In the present work methods and results are presented for a statistical determination of permeation in the N-5 strata of the Berozov GKM, undertaken to determine both the coefficient of variation and the degree of heterogeneity in permeation. Log-normal curves for permeation distribution were derived and relationships between the capture coefficient displaced formation gas and ratio of crude gas in producing wells employing various methods of the cycling process were established. Figures 2; references 4 (Russian).

UDC 622.276.031.011.436:536.5

**Effect of Temperature on Formation Gas When Working an Oil Stratum by Flooding**

907M0141F Kiev NEFTYANAYA I GASOVAYA  
*PROMYSHLENNOST*  
 in Russian No 1, Jan-Mar 90 (manuscript received 4 Apr 89) pp 31-33

[Article by G. V. Klyarovskiy, R. V. Mysevich, and B. G. Parakhin, Ukrainian Professional Oil SRI]

[Abstract] Cyclic flooding of an oil stratum through injection wells causes a drop in formation temperature, which rises again after termination of the flooding. In the present work a method was developed for computing the change in both volume and pressure of a geological formation system as functions of temperature. The thermobaric effects resulting from flooding operations were demonstrated to be insignificant in the case of Dolinsk oil field injection wells.

UDC 622.276.1/4001(477.8)

**Forced Recovery of Liquids at Late Stage of Oil Field Development**

907M0141g Kiev NEFTYANAYA I GASOVAYA  
PROMYSHLENNOST in Russian No 1, Jan-Mar 90  
(manuscript received 1 Feb 89) pp 33-34

[Article by V. I. Kirinyachenko, G. G. Gimer, and V. N. Silyava, TsNIL, "Ukrneft" Production Association]

[Abstract] Formation pressure has been sustained by water injection at the middle menilite, lower menilite, and eocene strata at the Gvizdetsk oil field since 1967, 1983, and 1984, respectively. All three strata have similar collector parameters, viz. mean porosity, 10.4 percent; permeability, 0.012 microns<sup>2</sup>; initial oil saturation, 74 percent. Being in the final stage of development, the oil field manifested low output at high water injection. Thus in Jul-Aug 85 forced liquid recovery was initiated at all three places. Gas lifting raised mean liquids output by 32 percent and crude oil by 31.5 percent. Figure 1.

UDC 622.276.5:53.08

**ASB-10 Optical Microprocessor Crude Oil Moisture Meter**

907M0141H Kiev NEFTYANAYA I GASOVAYA  
PROMYSHLENNOST in Russian No 1, Jan-Mar 90  
(manuscript received 1 Feb 89) pp 34-36

[Article by M. P. Yeselson, A. Z. Krikun, and V. A. Kuchernyuk, Ukrainian State Design SRI Petroleum]

[Abstract] Water content is a standard quality control criterion of crudes earmarked for laboratories or refineries. A moisture meter, developed at the title institute, employs pre-filtered infrared light (1.95 microns) and a microchip. The device facilitates automation of moisture determination with lower energy and reagent requirements, and is independent of the properties of the crude. Figure 1.

UDC 655.61.033.52

**Determination of Hydrogen Sulfide and Mercaptans Content in Crude Oil**

Kiev NEFTYANAYA I GASOVAYA  
PROMYSHLENNOST in Russian No 1, Jan-Mar 90  
(manuscript received 23 Aug 90) pp 36-37

[Article by Ye. Yu. Chardymskaya and O. M. Smirnova, Petroleum SRDI, Volgograd]

[Abstract] Hydrogen sulfide and mercaptans are corrosive and toxic ingredients of crude oil and gases, and determination of their contents directly at an exploratory well-head is of foremost significance. The sector on stratum crudes and gases of the title institute has developed a colorimetric method and device for determining these components. A sample of the head fraction is combined with a carrier gas and passed through a gas analyzer (indicator tube) where the mercaptans react with copper acetate to form yellow colored mercaptides. Hydrogen

sulfide reacts with lead acetate to form dark brown lead acetate. Both products are then determined colorimetrically. Figure 1; references 2 (Russian).

UDC 665.642.4.096-5.662.747

**Production of Vanadium Concentrates As Result of Deep Refining of High-sulfur Oils of Tatariya**

907M0313A Moscow KHIMIYA I TEKHOLOGIYA  
TOPLIV I MASEL in Russian No 8, August 1990  
pp 2-5

[Article by D. M. Soskind, R. N. Diyashev, V. V. Gribkov et al.; Scientific Research Institute of Petroleum Processing; Tatar Scientific Research and Design Petroleum Institute; All-Union Petroleum Scientific Research Geological Prospecting Institute; Moscow Region Petroleum Plant All-Union Scientific Research Institute of Petroleum Processing]

[Abstract] A quantitative assessment of deep refining of oils which are unique in the metal content in them involved the study of Nurlat and Bureya petroleums which are among the richest in the world in vanadium content. Tables presented: 1- list of petroleums and bitumens with extremely high vanadium content, 2- properties and composition of petroleums and their residues 500°C, 3- results of thermal-contact cracking of residues (hydrone), 4- characteristics of liquid distillates of direct distillation of petroleum and thermal contact cracking of hydrone, 5- characteristics of the cokes formed. Data presented in the tables were described and discussed. The study confirmed the possibility of complex deep refining of Tatariya petroleums which are rich in vanadium but which are improperly exploited. For each 1 million tons of Nurlat petroleum extracted, nearly 1000 tons of vanadium pentoxide is lost while a substance a hundred times more harmful than carbon monoxide or sulfur monoxide enters the atmosphere. These petroleums must not be extracted until appropriate means of processing them is available. References 5: 2 Russian; 3 Western.

UDC 665.642.4

**Deepening Petroleum Refining With Aid of Thermal Processes**

907M0313B Moscow KHIMIYA I TEKHOLOGIYA  
TOPLIV I MASEL in Russian No 8, August 1990  
pp 5-6

[Article by A. F. Makhov, A. I. Stekhun, R. N. Gimayev et al.; Bashkir Scientific Research Institute of Petroleum Processing, Novo-Ufa Petroleum Processing Plant, Bashkir State University]

[Abstract] The success of the Novo-Ufa Petroleum Processing Plant in refining high-boiling petroleum residues by delayed coking and thermal cracking was described and discussed. Experience gained from operation of the delayed coking process made it possible to increase the yield of large-piece coke, increase the inter-repair cycle up to 240 days and increase energy efficiency. Distillate

cracking-residue was used as coking raw material. Refinement of this residue with coke produced liquid products (nearly 50 percent). All coking distillates were used to produce commercial products. Fitting out of one of the thermal cracking devices with a unit for vacuum distillation of the tar oil cracking residue increased productivity of the equipment 1.5 times. One of the thermal cracking devices functioned, after reconstruction, in a tar oil viscosity breaking regime and the residue was used in furnace fuel oil. The wide use of thermal processing of heavy residues made it possible to intensify petroleum refining, to master production of low-ash electrode coke from sulfurous petroleums and to increase reserves of production of high quality motor fuels and other products. Use of the thermal processes increased the depth of refining at the plant to 65.5 percent. References 10 (Russian).

UDC 665.656.2.66.037.3

**Wastes of Butane-butylene Fraction as Raw Material For Production of Methyl-tert-ether**

907M0313C Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 8, August 1990 pp 7-8

[Article by P. N. Borutskiy, I. A. Volkov, V. Yu. Georgiyevskiy et al.; NPO "Lenmeftrekhim"]

[Abstract] Two procedures for refining waste butane-butylene fraction in order to increase production of high-octane components were examined. They are sulfuric acid alkylation of n-butylene by isobutane and geometrical isomerization of n-butlenes in isobutylene with subsequent refining into methyl-tert-butyl ether. Methyl-tert-butyl ether greatly exceeded alkylate as a high-octane component but isobutane found in the initial fraction may serve as alkylation raw material. Therefore, the proposed processes must be selected in terms of conditions of a specific plant. Block diagrams of the process of geometrical isomerization of n-butlenes and of production of methyl-tert-butyl ether from the butane-butylene fraction of catalytic cracking with isomerization of n-butlenes into isobutylene were presented, described and discussed. Use of geometrical isomerization of n-butlenes made it possible to increase methyl-tert-butyl ether production up to 168,000 tons per year, with reduction of production cost from 164.2-149.6 rubles per ton. The increase of methyl-tert-butyl ether production made it possible to expand the assortment of commercial gasolines; to increase profits from their sale and to increase the annual economic impact in comparison to that from the alkylation process. Figures 2; references 11: 10 Russian; 1 Western.

UDC 678.742.2.004.8

**Converting Ethylene-containing Gas—Waste of Polyethylene Production**

907M0313D Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 8, August 1990 pp 8-9

[Article by I. V. Popik, V. Ye. Leonov, B. K. Nefedov et al.; State Scientific Research Institute Methanol Project,

All-Union Scientific Research Institute of Petroleum Processing, Severodonensk Industrial Association "Azot"]

[Abstract] A search for efficient and ecologically safe methods of converting ethylene-containing gases into motor fuel components used H-ZSM type, high-silicon, zeolite-containing catalysts. Studies were performed on a flow-type device at a pressure of 0.2-0.4 MPa with flow rate of ethylene-containing gases of 60-100 liters/hr. Four catalysts were used. The study confirmed the possibility of converting ethylene-containing gases into motor fuel components on the zeolite-containing, high-silicon catalysts used with catalyst 4 being most efficient. Tests of the liquid hydrocarbons showed their suitability as motor fuel components. An experimental-industrial device for converting gaseous wastes of polyethylene production into motor oil components was developed. Figures 2; references 8: 3 Russian; 5 Western.

UDC 015.23:66

**Separator-Condenser For Catching Vapors of Low-boiling Liquids**

907M0313E Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 8, August 1990 pp 15-16

[Article by A. A. Kolesnik, A. B. Sergeyev, Yu. A. Golovastikov and N. A. Nikolayev; Kazan Construction Engineering Institute, Kazan Start-up and Adjustment Administration of "Orgneftekhimzavody" Trust, Ryazan Petroleum Processing Plant, Kazan Chemical Engineering Institute imeni S. M. Kirov]

[Abstract] A vortex separation-contact device with an autonomous cold-supply system for intensifying catchment of gasoline vapors on an industrial device was developed and tested. The device is placed after a vacuum pump on the line for blowing-off the solvent from the DF-11 dope. Freon-22 served as a heat carrier. A block diagram of the device was presented, described and discussed. Chromatographic analysis of the dry part of the gas revealed the degree of purification of the blow-off gas and the balance method was used to determine the amount of the liquid part of the condensate at the intake and that trapped in the device. The vortex-separation-contact device operated steadily regardless of liquid and gas loading. The degree of gasoline recovery exceeded 90 percent in all regimes, which is twice as high as the yield from reflux condensers. The compact device provided savings of 2.6 tons of gasoline per diem. The patented device exceeds domestic and foreign analogs in functional capacity. Figure 1; references 4 (Russian).

UDC 662.758:658.567

**Detergent Properties of Alcohol-ether Components of Automobile Gasolines**

907M0313F Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 8, August 1990 pp 17-18

[Article by O. P. Lykov, T. P. Vishnyakova, S. V. Zavyalov et al.; MING imeni I. M. Gubkin, All-Union Scientific Research Institute of the Petroleum Industry]

[Abstract] A study of detergent properties of automobile gasoline components which possess anti-icing properties was described and discussed. The components were produced on the basis of still residues of alcohols production. Gas-liquid chromatographic studies showed that these residues are a complex mixture of oxygen-containing compounds. They contain alcohols C<sub>4</sub>-C<sub>8</sub>(16-39 percent), butyrate (21-23 percent), butyryls (14-16 percent), simple butyl ethers (about 4 percent) and monoglycol ethers (up to 14 percent) and other compounds. The detergent effectiveness of the components differed somewhat. They produced good dissolving and solubilizing action and were quite effective in preventing formation of deposits. Addition of small quantities of these oxygen-containing compounds to gasoline helped to keep carburetors clean. Special detergent additives were not required when they were added to gasoline. References 3 (Russian).

UDC 665.761/765

#### Effect of Metallic Additions on Properties of Soap-free Lubricants

907M0313G Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 8, August 1990 pp 21-22

[Article by M. B. Chepurova, R. I. Kobzova, M. B. Bakaleynikov and S. V. Rastatuyeva; All-Union Scientific Research Institute, Petroleum Industry]

[Abstract] A comparative study of the effect of brass and copper formate additives on lubricants based on perfluoropolyesters, loaded with graphite, silica gel, ammeline or molybdenum disulfide was performed and discussed. The greatest load carrying capacity appeared in lubricant loaded with graphite. Anti-wear properties of all lubricants were approximately the same at loading of 200 H. Evaporability of silica gel and ammeline was at the same level. Additives used were brass powder and copper formate in a quantity of 10 percent and 5 percent (of the mass) respectively. Addition of brass powder to silica gel lubricant decreased its heat resistance but did not affect heat resistance in ammeline lubricant at 200°C but decreased it somewhat at 250°C. Heat resistance of graphite lubricant was much lower than that of silica gel lubricant and ammeline lubricant. All lubricants with copper formate additive broke down in the first hours of heating at 200°C with mass loss of more than 80 percent. The load-carrying capacity of silica-gel and ammeline lubricant can be increased significantly by addition of brass powder or copper formate; these additives did not affect the properties of the graphite lubricant. Figure 1; references 2 (Russian).

UDC 665.662.34

#### Effect of Surfactants on Phase State of Oil-Phenol System

907M0313H Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 8, August 1990 pp 27-28

[Article by R. G. Yaushev, R. Z. Safiyeva and Kh. G. Mindiyarov; Novo-Ufa Petroleum Production Plant, Bashkir State University]

[Abstract] Investigation of the effect of polymethylsiloxane (PMS-200A) on the phase state of an oil-phenol system on the basis of data of translation diffusion of molecules of the solvent and solvates obtained by pulsed nuclear magnetic resonance involved a study of a distillate of the butyric fraction of 350-420°C from a mixture of Tuymaz and Western Siberian petroleums. The nuclear magnetic resonance study showed that, at an optimal concentration of PMS-200A, the selective properties of the extractant intensified, the effectiveness of purification of the butyric fractions by phenol increased because of more complete extraction of polar resinous, hetero- and heavy aromatic compounds in the extractive phase. The nuclear magnetic resonance method can be used to study processes of solvation and intermolecular interaction directly in a phenol solution of the oils. Results of selective purification may be predicted on the base of the data obtained. Figures 3; references 5 (Russian).

UDC 691.16:541.64

#### Concerning Dependence of Viscosity of Mixtures of Petroleum Products on Composition

907M0313I Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 8, August 1990 pp 28-29

[Article by I. M. Kanevskiy, A. N. Aminov and V. V. Vasilyeva; Yaroslav Polytechnical Institute]

[Abstract] Proposal of a possible cause of appearance of extremes predominating for mixtures in which at least one of the components is a crosslinked liquid involved a study of the shear flow of mixtures of some petroleum products used as binding materials. Mixtures were prepared by intense mixing at 180-200°C and studies were performed on a rotational viscosimeter "Rheostat-2" at 30-150°C. Thermostating varied from 20 minutes at maximum temperature to several hours at minimum temperatures. Appearance of minima on the curve of dependence of viscosity on the composition of a mixture was attributed to imposition of 2 factors: changes of viscosity of the dispersion medium and changes of the volumetric percent of the dispersed phase with antibatic dependence of them on the composition. Figure 1; references 9 (Russian).

UDC 543.422.25.665.76

#### Determination of Means of Refining Natural Hydrocarbon Raw Material by Method of Nuclear Magnetic Resonance Spectroscopy

907M0313J Moscow KHIMIYA I TEKHOLOGIYA TOPLIV I MASEL in Russian No 8, August 1990 pp 30-31

[Article by D. F. Kushnarev, T. V. Afonina, A. A. Ageyenko and L. G. Treneva; Institute of Petrochemical and Carbonchemical Synthesis at Irkutsk State University, Yakut Scientific Research and Design Institute of the Diamond Extraction Industry]

[Abstract] Nuclear magnetic resonance spectroscopy <sup>1</sup>H and <sup>13</sup>C was used to determine structural characteristics of

petroleum and gas condensate of the Spredne-Botuobinskiy and Irelyakskiy deposits of Yakut ASSR respectively, and also their fractions for purposes of determining means of refining them and for preliminary assessment of operational properties of the basic products. Nuclear magnetic resonance analysis showed that light fractions of the petroleums and gas condensate contain predominantly paraffin carbohydrates of a low degree of branching. Naphthene and aromatic hydrocarbons were present in small quantities. At low octane number (48-50) of the gasoline fractions of the petroleum and gas condensate, the diesel fuel fractions had a satisfactory cetane number (38-42). Residues of the petroleum and gas condensate had a high degree of aromaticity and high dergees of condensibility of aromatic rings and branching of their substituents. The methods of nuclear magnetic resonance of <sup>1</sup>H and <sup>13</sup>C spectroscopy provided an easy, sufficiently accurate method of determining means of refining hydrocarbon raw material of new deposits. References 6; 5 Russian; 1 Western.

UDC 661.872:66.094.18

### Hydrogenation of Heavy Petroleum Residues in the Presence of Binary Catalysts

917M0019C Alma-Ata IZVESTIYA AKADEMII NAUK KAZAKHSKOY SSR: SERIYA KHIMICHESKAYA  
in Russian No 4, Jul-Aug 90 (manuscript received 24 May 89) pp 41-44

[Article by B.T. Yermagambetov, M.I. Baykenov, V.A. Khrupov, V.A. Mitrev, and A.Ya. Chen, Institute of Organic Synthesis and Coal Chemistry, KazSSR Academy of Sciences, Karaganda]

[Abstract] Iron-based catalysts are widely used in producing synthetic liquid fuel. Others have researched the catalytic activity of sulfide minerals, and the iron sulfides pyrite and pyrrhotite have been found to increase the degree of coal conservation. The study reported herein examined the integrated use of iron-containing compounds (wastes from metallurgical production) as an active additive to the commercial catalyst STK-1. A new catalytic system termed AB was created. It was prepared by mechanically mixing the two components. The "A" component consisted of metallurgical wastes with the following chemical make-up (mass %): Fe, 50.85; S<sub>tot</sub>, 32.84; WO<sub>3</sub>, 0.02; and Cu, 0.5. The "B" component consisted of commercial carbon oxide conversion catalyst comprising 88% Fe<sub>2</sub>O<sub>3</sub> and 8% Cr<sub>2</sub>O<sub>3</sub>. Bitumen from the oil refinery in Pavlodar was used as the starting material. The experiments were conducted in a rotary autoclave (capacity, 0.5 l; rotation speed, 60 rpm) in the temperature range from 400 to 450°C under an initial carbon oxide pressure of 2.0 to 8.0 MPa and with an operating pressure of 16.0 to 24.0 MPa. The following materials were used: 25 g bitumen, 15 g H<sub>2</sub>O, and 1.25 g AB. The experiment lasted 90 minutes. The gaseous phase was analyzed by gas-liquid chromatography, and the solid phase was isolated and subjected to x-ray phase analysis. Magnetite, siderite, pyrrhotite, and graphite crystalline phases were established. It was discovered that the maximum hydrogenation product was achieved with an A:B ratio of 1:4. As

the ratio increases, the yield of hydrogenation product decreases. The conversion of carbon oxide and yield of the fraction also decreased up to 300°C, which was likely due to a decrease in the reaction of the water gas. The optimum yield of liquid products lay in the pressure interval from 4.0 to 6.0 MPa, and the optimum temperature range was from 420 to 425°C. Figure 1; references 9: 2 Russian, 7 Western.

UDC 661.872:66.094.18

### Optimizing the Parameters of the Hydrogenation of Heavy Petroleum Residues by Using CO and H<sub>2</sub>O

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[Article by B.T. Yermagambetov, M.I. Baykenov, V.A. Khrupov, A.Ya. Chen, and A.L. Lapidus, Institute of Organic Synthesis and Coal Chemistry, KazSSR Academy of Sciences]

[Abstract] The process of hydrogen modification of heavy petroleum raw material entails increasing the H/C ratio by either adding hydrogen or else by removing carbon. The processes of catalytic and noncatalytic hydrogenation, catalytic cracking, and thermal splitting have been developed to lighten heavy petroleum residues. Hydrogenation processes are among the priority directions in intensifying refining. The main reason for the high cost of catalytic processing of bitumens is the rapid deactivation of the catalyst by the heavy metals contained in petroleum residues, hence the need to replace the catalyst and hydrogen by cheaper sources. In view of this, a process has been developed to hydrogenate heavy petroleum residues by using CO and H<sub>2</sub>O and synthesis gas in the presence of single-use catalysts. This article describes the attempt by the Pavlodar Oil Refinery to use CO and H<sub>2</sub>O to optimize the process of hydrogenation of heavy petroleum residues (bitumen). The optimization process was based on the factorial experiment-planning method and the MM PZO-87 computer program. The experiments conducted during the project were performed in an autoclave with a capacity of 0.5 l, a heating rate of 10°/min, and a rotation speed of 80 rpm. The autoclave was cooled by immersion into liquid nitrogen. The bitumen used contained the following (%): A°, 1.64; C, 85.35; H, 10.6; S<sub>tot</sub>, 3.5. It also contained 310 g/t vanadium and 70 g/t nickel. The following parameters were considered as factors in planning the experiment: temperature (X<sub>1</sub>), initial pressure of the carbon oxide (X<sub>2</sub>), contact time (X<sub>3</sub>), catalyst consumption (X<sub>4</sub>), and water consumption (X<sub>5</sub>). During the experiment portion of the study, 30 g bitumen, catalyst, and water were loaded into the autoclave. A specified amount of carbon oxide was added and heated the required amount of time. The autoclave was then cooled to room temperature, and the residual gas was measured on a GSB-400 gas counter and subjected to gas-liquid chromatography on an LKhM-8MD gas-liquid chromatograph. Comparison of the results obtained on the basis of the computer program and the

experimental data confirmed the adequacy of the mathematical description of the process and the possibility of forecasting the process conditions. Using the computer program made it possible to determine the optimal conditions for hydrogenation of bitumen (temperature, 425°C; initial CO pressure, 4 MPa; heating time, 90 minutes; and amounts of catalyst and water per 30 g bitumen, 1.5 g and 18 g, respectively). Figure 1, tables 3; references 4 (Russian).

UDC 66.074.3+661:18

### Methods of Scrubbing Sulfur-Containing Hydrocarbon Gases

917M0019E Alma-Ata IZVESTIYA AKADEMII NAUK KAZAKHSKOY SSR: SERIYA KHMICHESKAYA  
in Russian No 4, Jul-Aug 90 (manuscript received 12 Mar 90) pp 75-84

[Article by N.R. Bukeykhanov, K.R. Shayakhmetova, S.Zh. Zhardemgaliyev, and R.Yu. Kurochkina]

[Abstract] In the past few decades, the increasing shortage of carbon raw materials necessitated the commercial

assimilation of a number of oil and gas deposits containing hydrogen sulfide and a number of other sulfur compounds. The content of hydrogen sulfide and sulfur-containing compounds in oil and gas deposits varies widely, from 50 to 90%. At the same time, scrubbing out sulfur-containing compounds, specifically hydrogen sulfide, carbon sulfoxide, carbon sulfide, and mercaptans, has become an ecological imperative. This article reviews various methods of scrubbing sulfur-containing hydrocarbon gases with a high content of sulfur compounds that have been used in the USSR and abroad. The chemical and/or physical essences and pros and cons of various processes for primary scrubbing of sulfur-containing hydrocarbon gases are covered. These include alkaline scrubbing, chemisorption by alkanolamines, monoethanolamine and diethanolamine processes, a methyl-diethanolamine process, physical sorption-based processes, combined processes based on the principle of a physical solvent and chemisorbent, and adsorption-oxidation scrubbing. Also examined are processes of converting hydrogen sulfide into elementary sulfur and aftertreating the gases exiting Klaus units. Sixteen such processes are examined. Figures 2, tables 2; references 15: 6 Russian, 9 Western.

UDC 661.728.014/017:677.463

**Properties of Viscose Produced From Modified Cellulose**907M0234A Moscow *KHIMICHESKIYE VOLOKNA*  
in Russian No 2, Mar-Apr 1990 pp 32-34

[Article by A. K. Stavtsov, A. P. Meleshkevich, V. M. Irkley et al.]

[Abstract] A study of some basic characteristics of viscose produced from radiation-modified cellulose involved a comparison of properties of solutions obtained from sulfite standard cellulose and radiation-modified cellulose. Viscosity of viscose obtained from radiation-modified cellulose was lower than that of standard cellulose. Criteria of cross-linking and maximal Newtonian viscosity of viscose from radiation-modified cellulose were lower than these indicators in standard viscose and the change of these indicators with time was insignificant. Viscose from radiation-modified cellulose had higher coagulation resistance and physico-chemical homogeneity than standard viscose of analogous composition. Figures 3; references 5: 4 Russian; 1 Western.

**Some Problems of Producing Super-strong and High-modular Organic Fibers**907M0234B Moscow *KHIMICHESKIYE VOLOKNA*  
in Russian No 2, Mar-Apr 1990 pp 34-35

[Article by G. I. Kudryavtsev]

[Abstract] A discussion dealt with the possibility of using existing fibers to make construction composition materials rather than pursuing development of new material. It suggested examination of the problem from its theoretical aspects and the level of technology. It discussed the major importance of the rigidity of the molecules for super-strong, high-modular fibers and presented some findings on this matter. There has been a major breakthrough in producing high-grade fillers for construction of plastics in terms of the modulus of elasticity but no such breakthrough has appeared in relation to strength. The role of supermolecular organization of fibers in the general mechanism of failure is important. The achievements in this area and the problems still existing were discussed. The article recommended the application of efforts of chemists and technologists to create a technology for forming super-strong fiber. References 6: 4 Russian; 2 Western.

UDC 677.027.625.16(043.3)

**Spandex Threads With Decreased Combustibility**907M0234C Moscow *KHIMICHESKIYE VOLOKNA*  
in Russian No 2, Mar-Apr 1990 pp 37-38

[Article by M. A. Seredina and M. A. Tyuganova]

[Abstract] A study of the use of some halogen-containing compounds, especially organic halogen compounds, to retard combustion of spandex threads involved treatment of the threads with chloroendic acid, tetrachlorophthalic acid, and tetrabromophthalic acid. The threads were

treated with aqueous solutions of the acids at pH 2, 80°C and modulus of the bath 40. The threads were washed off with water up to a neutral reaction of the wash waters and dried at 70-80°C. Fire protection properties were assessed by the oxygen index. The study showed the possibility of producing spandex threads with decreased combustibility after use of these acids. Tetrachlorophthalic acid was the most effective. The spandex thread with decreased combustibility may be recommended for use in consumer goods in pure form and in a mixture with other chemical fibers. References 2 (Russian).

UDC 677.494.675.126.621.384.5

**Effect of Glow Discharge on Formation of Surface Layer of Polyacrylamide Fibers in Presence of Organophosphoric Compound**907M0234D Moscow *KHIMICHESKIYE VOLOKNA*  
in Russian No 2, Mar-Apr 1990 pp 38-40

[Article by S. V. Shapovalov, A. A. Kalachev, M. A. Tyuganova et al.]

[Abstract] Investigation of the interaction of a glow discharge with polycaproamide fibers impregnated with a non-volatile, nonreacting liquid involved a study of a complex thread (3.3 tex). The impregnating liquid was the product of copolycondensation of bis(dichloroethyl)vinylphosphonate and dimethyl ether of carboxyethylmethylphosphinic acid. The polyacrylamide fiber, impregnated with a 15 percent solution of the acid in chloroform, was dried at 50°C in a vacuum drier. Plasmochemical treatment of the impregnated fibers immobilized the impregnating agent. Contact of the glow discharge plasma with the impregnated material created migration processes of the impregnating preparation on the fiber surface and within it. The conditions of the discharge determined the intensity and the relationship of these processes. References 9: 5 Russian; 4 Western.

UDC 677.027.625.16(043.3)

**Characteristics of Thermooxidative Decomposition and Combustion of Fabrics From Mixture of Fibers**907M0234E Moscow *KHIMICHESKIYE VOLOKNA*  
in Russian No 2, Mar-Apr 1990 pp 40-42

[Article by A. Ya. Ivanova, M. A. Seredina, M. A. Tyuganova et al.]

[Abstract] A study of basic regularities of thermooxidative decomposition of fabrics consisting of a mixture of cellulose fiber and thermo-resistant fiber based on poly-n-phenyleneterephthalamide and from a mixture of cellulose fiber and fiber formed from a mixture of poly-n-phenyleneterephthalamide and polyacrylamide was carried out to create material with high fire-protection properties. Introduction of up to 12 percent (mass) of polyacrylamide into fiber from a mixture of poly-n-phenyleneterephthalamide and polyacrylamide greatly improved fire-resistannce of the fiber. Differences in the

mechanism of fire-protection action of a phosphorus-containing fire retardant for materials made from a mixture of cellulose fiber and fabrics made from poly-n-phenyleneterephthalamide and polacrylamide were established. Figures 3; references 2: 1 Russian; 1 Western.

UDC 677.494.745.32:677.027.623.582

**Optical Whitening of Freshly-formed Polyacrylonitril Fibers**

907M0234F Moscow KHIMICHESKIYE VOLOKNA  
in Russian No 2, Mar-Apr 1990 pp 42-43

[Article by I. N. Bykova, I. K. Gomzina, R. A. Salova and K. K. Strelnikova]

[Abstract] Experimental data showing the possibility and advisability of using a developed optically whitening substance for optical whitening of freshly formed nitron D fiber were presented and discussed. A liquid form of the whitener was compared to 2 of the best optically whitening substances (Khostalyuks and Blankodor). Use of the new whitener significantly increased the degree of whiteness of nitron D. Whiteness of the fiber increased with the increase of whitener concentration in the bleaching vat but decreased after reaching a peak. Other studies showed the whitener's superiority for whitening freshly-formed polyacrylonitril fiber by the continuous method. References 2 (Russian).

UDC 677.494.743.41.014.8:543.422

**Structural Changes in Fibers and Films From Polytetrafluorethylene After  $\gamma$ -irradiation**

907M0234G Moscow KHIMICHESKIYE VOLOKNA  
in Russian No 2, Mar-Apr 1990 pp 45-46

[Article by B. M. Tarakanov and O. Yu. Nikodimov]

[Abstract] Examination of the effect of  $\gamma$ -irradiation on the structure and properties of oriented polytetrafluorethylene involved the study of polytetrafluorethylene fibers and oriented at 370 K films produced by compression and sintering mark F-4D dispersed polytetrafluorethylene.  $\gamma$ -irradiation of polytetrafluorethylene fibers and oriented films caused shrinkage of the samples studied and change of their length with an increase of the absorbed dose. The effect of shrinkage and subsequent elongation of the samples tested depended upon the state of the samples. Shrinkage increased after increase of the number of drafts and decreased as a result of preliminary annealing. The behavior of the oriented polytetrafluorethylene was attributed to changes of the molecular and super-molecular structure. Figures 2; references 9: 8 Russian; 1 Western.

UDC 678.742.2.033.01

**Controlling the Properties of Polymer Composites by Directed Selection of the Matrix**

917M0019F Alma-Ata IZVESTIYA AKADEMII NAUK KAZAKHSKOY SSR: SERIYA KHIMICHESKAYA  
in Russian No 4, Jul-Aug 90 (manuscript received 6 Feb 90) pp 89-91

[Article by Zh.Zh. Bayarstanova, G.V. Kurbangaliyeva, L.N. Imasheva, Z.Zh. Sakiyeva, and Sh.Ye. Yerenova, Chemical Sciences Institute, KazSSR Academy of Sciences, Alma-Ata]

[Abstract] The technique of modifying thermoplasts by adding mineral fillers is widely used to produce materials with specified properties. The properties of highly filled composites are determined by the make-up of the composite, the technology used to produce both it and the products made of it, and many other factors; hence the importance of studying the effect of fillers on the mechanical characteristics of composites. This study examines the effect of the properties of the matrix on the mechanical characteristics of disperse-filled composites based on different grades of low-pressure polyethylene. The test specimens consisted of standard cast specimens based on polyethylene composites filled with different mass percents (10, 20, 30, 40, and 50) of natural calcium carbonate. Special attention was paid to the composites' initial viscosity and melt flow index. The melt flow index was not found to exert a decisive effect on relative elongation during stretching. It was discovered that the breaking stress during stretching increases as the true viscosity of the matrix increases and that the melt flow index may serve as a criterion for selecting polymers providing high deformation and strength characteristics in composites. It was further found that the dynamic strength of disperse-filled polymer composites can be controlled not only by introducing plasticizers but also by selecting the matrix. It was, for example, established that the impact strength of composites based on grade 273-74 low-pressure polyethylene is higher than composites based on grades 20908-040 and 277-73 low-pressure polyethylene with the same filler no matter what the calcite ratio. It was thus concluded that directed selection of the matrix may be used as a way of controlling the mechanical properties of disperse-filled polymer composites, particularly, to increase their resilience. Table 1.

UDC 541.125:546.185'33+546.62

**Reducing Polymer Phosphates by Metallic Aluminum, Magnesium, and Zinc**

917M0019G Alma-Ata IZVESTIYA AKADEMII NAUK KAZAKHSKOY SSR: SERIYA KHIMICHESKAYA  
in Russian No 4, Jul-Aug 90 p 92

[Article by V.A. Sinyayev, T.D. Vecherkina, and G.A. Tokseitova]

[Abstract] This concise communication reports a study of the reaction of metallic aluminum, magnesium, and zinc with cyclotrisodium phosphate, which is among those

polymer phosphates possessing the highest oxidation activity of that type of compound. The study has established that the reaction begins close to the cyclotriphosphate melting point and is accompanied by intense oxidation of the metals. The reaction is found to yield elemental phosphorus and binary sodium phosphates, i.e., multivalent metals with an impurity among the phosphates (aluminum, magnesium, or zinc). The depth of the process is found to depend on the nature of the metal, the temperature, and the concentration of reagents and duration of their contact. The yield of elemental phosphorus in all of the cyclotrisodium phosphate-metal systems studied did not exceed 10% of the total amount of phosphorus in the cyclotriphosphate. References 2 (Western).

UDC 677.494.745.32.021.125.2

### Diffusion Processes During the Formation of Polyacrylonitrile Fibers

917M0021A Moscow KHIMICHESKIYE VOLOKNA  
in Russian No 5, Sep-Oct 90 pp 6-8

[Article by E.R. Aliyeva, Yu.P. Kozhevnikov, V.A. Medvedev, and A.T. Serkov]

[Abstract] Precipitation of the polymer is the main stage in the production of polyacrylonitrile fibers from solutions when using the wet method. Its kinetics are determined by two factors. The first is the nature of the resultant supermolecular (morphologic) structure and the respective set of the fiber's user properties, and the second is the stability of the process, which has a significant effect on the process equipment, formation of wastes, and ecological conditions. The polymer precipitation process proceeds by substituting a solvent for the precipitating agent based on a molecular diffusion mechanism. The authors of the study reported herein examined the concentration of precipitating agent in the fiber formed from polyacrylonitrile solutions in a mixture of water and sodium rhodanide and determined the polymer's precipitation bands. Their goal was to obtain data that would be useful to process engineers responsible for selecting formation parameters and estimating the dimensions of process zones, specifically, the length of the zone in which the polymer stream would be solidified in the precipitation bath. They studied a 12% solution of a copolymer of acrylonitrile, methylmethacrylate, and itaconic acid in a 92:6:2 ratio with a molecular mass of about 45,000. A 12% NaSCN solution in water was used for the precipitation. They discovered that the coefficient of the diffusion of precipitating agent into the resultant polyacrylonitrile fiber equaled  $0.8 \cdot 10^{-5} \text{ cm}^2/\text{s}$  at 20°C. They also estimated the concentrations of precipitating agent in fiber formed from solutions of polyacrylonitrile in a water-rhodanide precipitation bath and estimated the duration of length of the zone of the polymer's precipitation in the stream of spinning solution. Figures 3; references 5 (Russian).

UDC 677.494:677.027.632.511.2:546.824-  
31.77.021.115.3

### Dispersability of Titanium Dioxide in Polar Media—Index of Manufacturability of Pigment With Matting of Fibers

917M0021B Moscow KHIMICHESKIYE VOLOKNA  
in Russian No 5, Sep-Oct 90 pp 11- 13

[Article by I.N. Bykova, I.M. Gomzina, S.I. Popryadukhina, and L.I. Bekkerman]

[Abstract] Producing synthetic fibers for use in textiles makes no sense unless matting is used. For nearly 50 years,  $\text{TiO}_2$  in an anatase modification has been used to eliminate the sheen and transparency of fibers and to increase their whiteness. Finely dispersed grades of  $\text{TiO}_2$  provide a higher degree of whiteness because small pigment particles ( $<0.15 \mu\text{m}$ ) scatter light blue light more strongly than red light. As the wavelength increases, the optimal size of the  $\text{TiO}_2$  particles increases as its diffusing power decreases. For this reason, those manufacturing  $\text{TiO}_2$  for use in producing chemical fibers produce  $\text{TiO}_2$  anatase with an average particle size of 0.3-0.4  $\mu\text{m}$  (with 90-95% of the particles being less than 1  $\mu\text{m}$  in size and only 2 to 3% being larger than 2-3  $\mu\text{m}$  in size). During storage and transport, the  $\text{TiO}_2$  undergoes partial agglomeration. As a result, it must be redispersed (i.e., the agglomerates must be broken down) so that the individual  $\text{TiO}_2$  particles are evenly distributed in the dispersing medium. Most domestic and foreign enterprises perform this necessary operation on special equipment. Since the dispersability of  $\text{TiO}_2$  in prepared suspensions as well as in the matted polymer depends primarily on the dispersability of the pigment itself, the dispersability of the pigment in the specific polar medium used in manufacturing a given fiber must be considered one of the main indicators of the manufacturability of  $\text{TiO}_2$  (for example, pigment is considered suitable for matting monacrylic fiber if its stability in acetone is at least 72%). One way of improving the dispersability of  $\text{TiO}_2$  is to modify its surface. Most commercial grades of  $\text{TiO}_2$  are distinguished by their type and amount of modifier rather than by their pigment itself. In view of the great importance of the interaction of the pigment and dispersing medium, the authors of the study reported herein examined the dispersability of eight different grades of  $\text{TiO}_2$  as a function of the type of modifying additives contained in the pigments and as a function of the medium. The following grades of  $\text{TiO}_2$  were studied: Kronos AD, Kronos AVF, LW-SU, TA-500, TA-300, ANR, A-RR2, and A-1. The modifying additives used were as follows:  $\text{TiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{P}_2\text{O}_5$ ,  $\text{ZnO}$ ,  $\text{Mn}^{2+}$ ,  $\text{K}_2\text{O}$ , and  $\text{Sb}_2\text{O}_3$ . As a result of their studies the authors strongly recommended the LW-SU, TA-300, and AHR titanium dioxide for use with polyester fibers; TA-500 for use with nitron-M; and A-RR2 and TA-500 for nitron-D and polyamide fibers. They also found that despite its inadequate self-dispersability, Kronos AVF remains unrivaled for matting polyamide fibers. Tables 2, references: 2 Russian, 3 Western.

UDC [628.33:66.081.3.32]:541.183.5

**Selecting Microporous Adsorbents for Waste Water Treatment**

*917M0021C Moscow KHIMICHESKIYE VOLOKNA  
in Russian No 5, Sep-Oct 90 pp 24-27*

[Article by Ye.I. Khodorov and V.V. Semerikova]

[Abstract] The increasing scales of manufacturing and the stiffening of sanitary standards have made it necessary to find more effective and economical ways of removing pollutants from waste waters and recycling the cleaned waters. One of the most promising methods for so doing is adsorption treatment (or aftertreatment) of process waste waters with low initial concentrations of pollutants, in which case other methods become economically unfeasible or even impossible. Activated charcoal-based adsorption cleaning of waste waters has become a very popular method in chemical process industries abroad. Wide-scale introduction of sorption methods for local waste water treatment in the USSR is being held back primarily by the shortage and expense of sorbents, especially activated charcoal. One promising way of developing the method of adsorption treatment and improving its technical and economic indicators is thus to find absorbents that are less expensive and more effective than activated charcoal. For this reason, the authors of this article undertook the task of developing an integrated approach to selecting micro-porous absorbents for use in waste water treatment. Because most of the costs associated with the technology of sorption treatment (50 to 80%) are incurred in obtaining the sorbent and only 5 to 25% of the total costs are related to recovering the sorbent, the authors focus solely the costs of replenishing the sorbent expended rather than on the costs associated with recovering sorbent. They calculate the cost of a prospective absorbent by using the equation  $C_{repl} = L_{rel}S_{sp}P_{opt}$ . In this equation  $C_{repl}$  is the cost of replenishing the absorbent used in the process;  $L_{rel}$  represents the relative losses of absorbent in the processes of adsorption, regeneration, reactivation, and transport;  $S_{sp}$  is the specific consumption of sorbent; and  $P_{opt}$  is the optimal price of the sorbent. They proceed to derive these quantities by using a series of equations. Next, they test their method by way of the example of three prospective carbon sorbents (IGI, AA, UM) for treating caprolactam-containing waste waters from the production of polyamide fibers. According to their calculations, the sorbents IGI and AA turn out to be the most promising. Tables 2; references 9 (Russian).

UDC 677.025.072

**New Yarn for Producing Knitted Fabrics and Knitwear**

*917M0021D Moscow KHIMICHESKIYE VOLOKNA  
in Russian No 5, Sep-Oct 90 pp 32-36*

[Article by A.V. Paramonov, candidate of technical sciences, and N.A. Grechukhina and M.Ye. Milyayeva, engineers, All-Union Scientific Research Institute of the Knitted Fabrics Industry, Moscow]

[Abstract] The work of the All-Union Scientific Research Institute of the Knitted Fabrics Industry with respect to using knitted fabric is proceeding in two directions. The first is developing new types of yarn and using it in a range of knitwear. The second is assessing the quality and product line capabilities of the new types of yarn with respect to raw material make-up, linear density, structure, color matching, and consumer properties. With regard to the the first direction, the institute has collaborated with the textile plant Krasnaya veta to develop a 100-tex cotton yarn (consisting of 70% cotton and 30% synthetic fibers 90 mm long) that is spun by a pneumatic method. The yarns have good processing qualities, are soft and pleasant to the touch, and result in top-quality finished products. The technology for manufacturing these yarns has been installed at the Krasnaya Veta Plant. The institute has also collaborated with the cotton-spinning mill to develop combined a new cotton and kapron yarn in a product line ranging from 17.6 to 31 tex. The basic 15.4-tex yarn consists of 15.6-tex complex kapron threads, 4.8-tex trilobal [transliteration], and 2.2-tex monokapron. Knitted fabric and knitwear made of the new yarn demonstrated good consumer qualities and proved to be soft, pleasant to the touch, and wear resistant. The All-Union Scientific Research Institute of the Knitted Fabrics is also collaborating with the Dedovsk Technical-Grade Fabric Production Association on a combined nitron and viscose yarn. Another new development on which the institute is working is a yarn that combines cotton-viscose and wool. In line with the recent trend abroad toward an increased use of flax in knitted fabric, the institute is conducting joint research on 72- and 50-tex yarns consisting of cotton (35%), viscose high-modulus fibers (35%), and flax fibers (20%). Production of this yarn has been set up at the Reutov Cotton Mill. Inexpensive flax fiber used in manufacturing packcloth is used in the new yarn. A line of cotton yarns with special effects is also being developed by the institute, as is a line of cotton and cotton-viscose yarns that are twisted with another viscose thread and a 20-tex improved-quality cotton yarn. Tables 4.

UDC 677.025.072:677.463+677.025.072.76

**Knitted Fabric Made of New Viscose Threads**

*917M0021E Moscow KHIMICHESKIYE VOLOKNA  
in Russian No 5, Sep-Oct 90 pp 37-38*

[Article by V.Ya. Biryukov, G.P. Kuzmina, and Ye.I. Telkova, engineers, All-Union Scientific Institute of the Tricot Industry, Moscow]

[Abstract] Because of their stylishness and comfort, glittering viscose threads have become increasingly popular for use in knitwear. To expand the range of viscose threads and create high-quality knitwear, the All-Union Scientific Research Institute of the Knitted Fabrics Industry has collaborated with a number of knitting factories and production associations to create new two-color viscose glittering threads. The new threads are distinguished by their interesting color effect, which is achieved by pneumatically joining two threads of different colors. Two such new threads (33.2 and 61 tex) were studied. The threads were found to have the following properties: specific

breaking load when dry,  $>98$  mN/tex; stretch, 18-25%; and cohesion (characterized by the number of slubs per meter),  $\geq 17$  for the 33.2-tex thread and  $\geq 10$  for the 61-tex thread. By using different combinations of white, gray, black, and yellow threads, the researchers were able to achieve a variety of interesting effects. Specifically, by combining achromatic colors with yellow and two achromatic (white and gray), they were able to obtain various tones simulating silver, copper, and bronze. Studies conducted to determine problems that might arise when the new threads are used on conventional textile equipment led the authors to recommend that the 61-tex thread be used on flat-rib machines to create effective color bands simulating metallic threads and on circular knitting machines to create structural color elements in knitwear. The pneumatically joined 33.2-tex threads were not found to cause any complications when used in making fabric or knitwear. From the standpoint of their physicomechanical properties, the fabrics developed meet the quality requirements stipulated in GOST 16486-83. Their shrinkage did not surpass the standard, i.e., it amounted to 5% lengthwise and ranged from 2.8 to 4.5% crosswise. The fabrics were also found to be suitable from standpoints of style and pleasantness to the touch. Table 1.

#### Using Polyurethane Threads With Increased Shrinkage in Hosiery and Sock Production

917M0021F Moscow KHIMICHESKIYE VOLOKNA  
in Russian No 5, Sep-Oct 90 pp 39-40

[Article by Ye.A. Galkina and G.P. Kuzmina, engineers, All-Union Scientific Research Institute of the Knitted Fabrics Industry, Moscow, L.V. Savinkova, VNIISP (expansion not given), and Yu.V. Fedyunin, Khimvolokno Production Association, Volzhskiy]

[Abstract] The problem of the adhesion of threads on bobbins is one of the factors preventing the wider-scale use of the domestically produced low-linear density polyurethane elastomer thread, which is used in producing hosiery and socks. While the effects of this problem have not yet been quantified, they have been implicated in complaints regarding the quality of such products. Research has shown that end breaks of threads because of adhesion during processing on automatic seamless hosiery machines occur from 8 to 20 times per kilogram of processed raw material (the norm being from 2 to 5 cases per kilogram). In an effort to reduce the adhesion of threads on a bobbin, the authors of this study investigated the possibility of using spandex polyurethane elastomer threads that have not been subjected to preliminary thermal fixation. Such threads are distinguished by their increased take-up, which reaches 15% as opposed to the normal 3 to 5%. Despite this fact, they still meet the existing specifications. Studies of the polyurethane elastomer spandex threads on automatic seamless hosiery machines revealed that end breaks occur 2.5 times less frequently in threads that have not been subjected to thermal fixation than in threads that have. Tests of the physicomechanical properties of the non-thermally fixed threads confirmed that the higher take-up of such threads does not reduce their quality because spandex is restricted by its loop structure. Furthermore, tests of pantyhose made with the threads in question had a

better stretch and a lesser residual deformation, i.e., better elastic properties. The tests results were later confirmed by the results obtained at a number of hosiery plants. As a result of the study, 8- and 15.6-tex non-thermally fixed polyurethane elastomer spandex threads were recommended for use in producing hosiery and socks. Tables 2.

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#### Artificial Fur Made of Polyvinylchloride Fibers

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in Russian No 5, Sep-Oct 90 pp 40-41

[Article by N.M. Ivanova, candidate of technical sciences and L.G. Faetonova and O.A. Kuznechik, All-Union Scientific Research Institute of Polyvinylchloride, Moscow]

[Abstract] The All-Union Scientific Research Institute of Polyvinylchloride has created a line of fabric-based artificial fur by using a napped covering of unbleached polyvinylchloride braided fiber (0.33 tex) produced by the Khimvolokno Production Association in Kustanay. The range of furs and the areas in which they may be used depend on the properties of the fiber. One such property, namely, heat shrinkage, determines the fur's external appearance and quality. For polyvinylchloride fiber, the optimal temperature for heat treatment in air is between 100 and 110°C for a processing time of 1 to 2 minutes, in which case the shrinkage amounts to 30%. When the treatment time is increased to 4 minutes, the shrinkage increases to 50%. Repeated heat treatment helps shrink the fiber even more and changes its physicochemical properties. Because of its high shrinkage properties, polyvinylchloride fiber can be used to create artificial fur with an underfur by using a napped yarn base (41 tex x 2) consisting of 33% unbleached polyvinylchloride and 67% dyed polyacrylonitrile. During the heat treatment process the polyvinylchloride shrinks significantly, thus forming an elastic underfur. For commercial use of such fur, however, it is necessary that dyed polyvinylchloride fiber be used. Otherwise the contrasting colors of the fibers in the napped covering will not result in a satisfactory appearance. To reduce this shortcoming and expand the range of dress fur, the authors of this study developed an artificial fur with an underfur that has a combined thread (65.6 tex) consisting of mixed yarn (33% unbleached polyvinylchloride fiber and 67% dyed polyacrylonitrile fiber) and a polyamide complex profiled thread (15.6 tex). The unbleached stichel of polyamide threads softens the difference between the colors of the fibers used. The fur was subjected to contour shaving to form a napped surface. The resultant product has high physicomechanical indicators, the napped loops remain secure in the face of a force of up to 130 cN, and the wear of the nap does not exceed 14% of the loss of the nap's mass. The artificial furs have improved heat insulation properties and have been approved for lining footwear. The institute also developed a polyvinylchloride-based artificial fur that meets fire safety standards for toys.

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**Structure and Properties of Conductive Threads**  
917M0021H Moscow *KHIMICHESKIYE VOLOKNA*  
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[Article by V.K. Smirnov and A.V. Kovarskiy, candidates of technical sciences, and V.A. Krasheninnikova and I.A. Martemyanova, All-Union Scientific Research Institute of the Textiles and Haberdashery Industry, Moscow]

[Abstract] The manufacture of conductive chemical fibers, filaments, and yarns for producing different products possessing electrophysical products has recently begun in the USSR and abroad. Textile products with electrophysical properties have a number of advantages over analogous products made of the conventionally used metal filaments with a small cross section. These advantages include high mechanical strength, stability in the face of multiple cyclic loads and bending, and greater convenience of processing. The shortcomings of such textile products include their low conduction, high specific resistance, the complexity of the processes entailed in metallizing them, and their aging during lengthy use and storage. Thus, the decision to use metal or conductive chemical fibers depends on the specific conditions under which a given product will be used. To shed more light on this subject, the authors of the study reported herein compared the structure and selected physicomechanical properties of conductive chemical filaments produced by different methods in order to determine the feasibility of using each of them. They studied the following: complex polyamide and glass filaments coated with conductive layers of carbon (carbon black), polypropylene filaments metallized by the electrochemical method, and aluminum-coated polyester strips with a cross section of  $0.5 \times 0.5$  mm coated that have been additionally coated with a polyester film to protect the strips from oxidation. A steel filament (diameter, 50  $\mu\text{m}$ ) coated with a protective nickel layer was used as a standard specimen. The specimens were studied by light and electron microscopy, on a Tsvik automatic tensile testing machine, and with an AVO-541 multimeter. The structure of the coatings of the chemical filaments tested was found to contain vacancies and separate, non-interconnected particles, which increases their specific electrical resistance and makes it impossible to recommend these filaments instead of metal filaments. The coatings of the metal filament studied also have microinhomogeneities reducing the functional properties of the products containing these filaments. The chemical filaments were, however, superior from a tensile strength standpoint. The test results were recommended as a starting point for planning studies to find ways of improving the chemical filaments examined. Figures 6, tables 1.

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**Effect of the Chemical Make-Up of Synthetic Fibers on the Fire Protection Properties of Mixed Materials**

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in Russian No 5, Sep-Oct 90 pp 45-46

[Article by A.Ya. Ivanova, candidate of technical sciences, and M.A. Tyuganova, doctor of chemical sciences, Moscow Technology Institute imeni A.N. Kosygin, Moscow]

[Abstract] The production of fire-protective synthetic fibers is a fairly recent development, hence the interest in developing new types of textiles from a mixture of synthetic and fire-protective cellulose fibers. In view of this interest, the authors of this study examined the laws governing the thermal oxidative breakdown of fabric made of a mixture of viscose and polyester fibers (67:33) and cotton and oxalon fiber (50:50) before and after fire-protective treatment. These types of fibers are distinguished by their capability of carbonization, and oxalon is distinguished by its elevated thermal stability. When both the viscose-lavsan and cotton-oxalon fibers are treated with T-2 antipyren, their cellulose component reacts with the fire retardant, whereas the synthetic fibers do not form a chemical bond with the antipyren. Before fire-protective treatment, the cellulose fiber in viscose-lavsan fabric affects the nature of the breakdown of the polyester component. After treatment, on the other hand, there is no effect. Evidently, the rapid carbonization of cellulose modified by antipyren T-2 eliminates the melting of polyester. At the same time, after fire-protective treatment of the fabric, the phosphorus-containing cellulose ether in the mixed material breaks down under conditions characteristic for an individual fire-protective cellulose fiber in an analogous process, in which case the thermolysis activation energy decreases along with the decomposition rate and temperature. The main feature of the thermal oxidative decomposition of polyester in the presence of phosphorus-containing cellulose was that the order of the thermal oxidative reaction was increased somewhat. The studies of the effect of oxalon revealed that adding this fiber changes the direction of thermolysis, with the amount of phosphorus-containing compounds changing into a gaseous phase decreasing markedly. Whereas more than 50% of the phosphorus in viscose-lavsan fabric moves into the gaseous phase during decomposition in the temperature range from 300 to 400°C, only 16-19% does so during thermolysis of material containing oxalon. The effectiveness of the action of T-2 antipyren was determined to be 5.57 for material consisting of a mixture of cotton and oxalon and 3.2 for material containing polyester. The authors thus confirmed the predominant role of the reactions occurring during the condensed phase of pyrolysis. Figure 1, tables 4; reference 1 (Western).

UDC 547.233:547.314.2

**Phosphorylation of 1-Ethynyl-1-Aminocyclohexane  
by Dialkyl Phosphorous Acids**

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[Article by S. K. Tukanova, B. Zh. Dzhembuyev and B.  
M. Butin, Institute of Chemical Sciences, Kazakh SSR  
Academy of Sciences, Alma-Ata]

[Abstract] A series of phosphorylations at room temperature in carbon tetrachloride of 1-ethynyl-1-aminocyclohexane by dialkyl phosphorous acids were carried out as part of a search for novel polyfunctional organophosphorus compounds. As a result, the following compounds were synthesized: O,O-dimethyl-, O,O-diethyl-, O,O-dipropyl-, O,O-dibutyl- and O,O-di( $\beta$ -chloroethyl)-N-(1-ethynylcyclohexane-1-yl) amidophosphate. References 1 (Russian).